

7N-24-CR
157061

MINUTES
ADVISORY COMMITTEE
ON STANDARDIZATION OF C-P TEST
METHODS AND SPECIFICATIONS LAGUNA HILLS, CA
MAY 3, 1988

for
Standardization of the Carbon-Phenolic and
Carbon-Carbon Materials and Processes
NASA Grant No. NAG8-545

Prepared for
National Aeronautics and Space Administration
George C. Marshall Space Flight Center, Alabama 35812

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August, 1988

(NASA-CR-183158) STANDARDIZATION OF THE
CARBON-PHENOLIC AND CARBON-CARBON MATERIALS
AND PROCESSES (Mississippi State Univ.)
116 p

N88-71439

Unclas
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MISSISSIPPI STATE
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MINUTES
ADVISORY COMMITTEE
ON STANDARDIZATION OF CARBON-PHENOLIC TEST
METHODS AND SPECIFICATIONS AT
LAGUNA HILLS, CA
May 3, 1988

PRESENT:	Bill Hall, Chairman	Miss. State University
	Linda Doss, Administrative Assistant	Miss. State University
	Ed Hemmelman	Fiberite, Winona
	John R. Koenig	Southern Research
	Eric Stokes	Southern Research
	Patrick Doan	Morton Thiokol
	Jon Weispfenning	Fiberite, Winona
	Lou Ann Fikes	NASA, MSFC
	Thomas A. Paral	Polycarbon, Inc.
	Don A. Beckley	U.S. Polymeric
	Scott Jackels	Fiberite, Winona
	Pat C. Pinoll	Lockheed Research
	Ben Neighbors	NASA-MSFC @ MTI
	Gary Hall	Morton Thiokol
	Ronald E. Higgins	Polycarbon, Inc.
	Pat McGill	Fiberite, Laguna Hills

Dr. Bill Hall, Chairman, called the meeting to order at 9:16, Tuesday morning, May 3, 1988. Dr. Hall gave a brief opening statement concerning the present ongoing contract that he was involved in with the Marshall Space Flight Center which is the "Standardization of the Carbon-Carbon and Carbon-Phenolic Materials and Processes", this was the initial three year integrity program which concludes this summer. The objective of that particular program, was to select a set of specifications for the materials and processing of carbon-phenolic composite materials which yield a predictable and reproducible product with desired properties. We have massaged the test methods and specifications for three years with minimal change in either set of parameters. The test methods are essentially the same as when we started three years ago, the specifications are the same.

In the second contract, it was decided to set-up an advisory committee which would be industry wide and this is that committee. The statement of work for this committee is to establish and administer an industry-wide advisory

committee that will meet twice a year to review, revise, and to/subtract from present specifications and test methods and make recommendations for evaluation of new test methods and specifications.

Dr. Hall emphasized that this committee was established for industry and this committee was established and recognized by Marshall and this is one of the things they will rely on when and if we make recommendations for changes. Dr. Hall encourages each committee member to take this appointment seriously and everyone participate to the maximum capability of their particular situation.

Dr. Hall stated that today's meeting would be a broad discussion of the purpose of this committee and the parameters this committee would like to initiate. Without further discussion, the floor was open for discussion.

John Koenig Referring to the view graph on "Materials Supplier Flow" which refers directly to either FM5055 or MX4926; are we particularly working against those two materials or is it more broadly carbon phenolic ...(Involvement)?

Bill Hall It is broad but we will use this as our basis to start with. You are well aware that NASA is looking for alternate materials.

Pat Pinoli I don't know of any action on the current RSRM program that is designed to replace the existing material; therefore, the alternate material is being driven by ASRM.

Bill Hall That is correct.

Pat Pinoli In that case, would we then have a charter to work on advanced materials? My perception of the current SPIP program primary objective is to advance our understanding of current materials and the secondary objective is to provide an alternate material in case the current material fails to meet future flight performance requirements. The ASRM program is encouraged to use more advanced materials and design.

Ben Neighbors My particular concern is what we are flying today compared with what we will be flying five or ten years from now. A lot of advancement can be made with what we are currently using.

Pat Pinoli The original criteria of this program was to only concentrate on current HPM nozzle materials.

Bill Hall The original criteria in the first program for evaluating the test methods, it follows of course you can't have specs unless you have good test methods, was:

1. Does the test method accurately measure the desired property?
2. Does this property yield useful information for the evaluation of the material to perform as required?

Those are the two criteria that we looked at when we evaluated the test methods under the original grant. How do we want to define "accurately measure the desired property"? For example, one of the test methods we looked at was the resin flow from the prepreg. One way to measure it, is that you cure a 4x4 square, scrape off the extruded resin and re-weigh it. There is another way of doing this. You use the same cured 4x4 square, but you use a template to cut-out a circular specimen which is exactly half the cross-section area, since it is said that resin flow is a radial phenomena. If that is true, then this would more accurately measure the resin flow.

Are we really interested in getting a different number? This would generate a different but higher number. Does it yield any more useful information?

John Koenig

I'm not sure. It makes an assumption that the flow of the phenolic resin is uniform in a radial method. I'm not sure that this is true.

Bill Hall

We need to establish a philosophy here. Are we interested in a pure absolute number or making sure that what we measure today is what we measured yesterday and the day before and it is the same material. What was originally established was that we had a nozzle material that worked, and the test methods and specifications were set-up so that the components we made today were the same components that we made yesterday and will be the same that we make tomorrow. In that sense, all that we are interested in is accurately measuring "Is it the same material?" Reproducibility or precision. For example, if you measure residual vols, you can get a number for 4 hours, 24 hours, or 48 hours, (in the dryer) they are all different numbers, but don't they give you the same information? You can then test it under vacuum and get another number.

Pat Pinoli

Your example clarifies the issues considerably. In the past, the relevancy, accuracy and precision of tests were unknown. Current studies however have greatly enhanced our understanding of these issues and with respect to the residual volatile test we have to decide whether water, permeability, total residual volatiles or % water are the critical properties we need to track.

John Koenig

The last example makes it an easy one. It is broken down into a two parameter test and it is very possible to alter those two parameters and get exactly the same results for very different materials. The parameters are the diffusion of moisture out and

the moisture content. From a quality point of view it is also a poor test, because if you are given the same value when the material is very different and from a material property or material understanding point of view, it is also a poor test.

Don Beckley

It is worth while in making the point that you are dealing with an acceptance test in a spec and you intend to run it on a repetitive basis, the purpose then is not to do research or trying to find out, but the purpose is to try and find out if this unit is the same as all the other units. At that point you are just saying I have fixed the test now the number has to fall within my previously defined history or I have a reason to be concerned that something is amiss. I feel that is the criteria that we have to keep in mind for the subject.

John Koenig

That is a key point and I presume that Don's statement is accepted; that we are to establish a quality test.

Bill Hall

I am almost sure this is what NASA wants out of this committee. I think Don is right in that we are not after research in this particular instance. What we want to do is to assure the safety, if at all possible, of the nozzle integrity as it is used.

John Koenig

I am quite willing to buy off on that definition although your question 2 is a key point ... towards the property as opposed to a response.

Bill Hall

I think we are going to have that problem over and over again.

Pat Pinoli

The committee should look very carefully before instituting these tests to determine if it can at all possible establish a test that is accurate and have looked at all the parameters that affect that test.

Bill Hall

How do you define accurate?

Pat Pinoli

I can appreciate the problem of defining an accurate test. By your definition, the current tag end residual volatile test is accurate since it meets your definition as a "quality" or comparative judgement test. I believe the current test, however, is not accurate with respect to measuring total residual volatile level in tag end specimens and does not provide permeability data which could be of equal value. The problem of residual volatile test accuracy can easily be resolved by defining what is the test designed to measure i.e., total volatile level, water content, permeability, etc. All of these factors are related to test temperature and a decision must be made as to a standard test temperature. My judgement on this issue is the test temperature should be high enough to induce complete condensation reaction and below pyrolysis temperature – 500°F. This approach brings science into the testing program as opposed to the current obsolete methodology. My argument with the current test is relevant to those of us that believe the high volatile level is bad and those that believe a high volatile may be good with respect to component behavior. Both views require an accurate assessment of residual volatiles in components. The current technique does not provide an accurate assessment of total residual volatiles at 325°F and is strongly influenced by specimen permeability, therefore a simple engineering judgement that the product is similar to previous production articles is erroneous. These should be accepted facts by all of us and to ignore the issues is unacceptable to me. We may not have the test bed verification data required to couple cause and effect but we do have the analytical capability to measure

these parameters for engineers to pass judgement on at a latter date. Improved analytical testing procedures are designed to diminish the potential for more ETM-1A surprises. The current testing program was obviously inadequate.

Jon Weispfenning

Don't you first have to establish what properties you are interested in first before you take off Once you find out what you want to test then you have to figure out what test methods you want to measure accuracy and reproducibility, the bottom line is to make something non-test method dependent/non-technique dependent as much as possible.

John Koenig

If you are non-technique dependent you are driven toward the problem. Another thing that will come out of question 2. For example, one of the quality control now opposed is the room temperature warp direction compression which is almost totally irrelevant in any ...

Bill Hall

I think the second statement there is just as important as the first.

Ed Hemmelman

Aren't we getting confused. We are talking about a specification and developing data. In my way of thinking the specification generates some properties that guarantees to you that the product you make today is the same product you made last week or the same product you qualify. I don't think we should be driven toward trying to get the engineering data necessarily out of the material we are trying to judge as some measure of quality.

John Koenig

I think that is going to be accepted when we accepted Don's statement that we are not attempting to get engineering data, but that it is important for us to assume that the QC technique

for each test that we choose is one that monitors a property, which is keyed to a response so that if that event changes we know that there is a very real reason to look further into the variation.

Don Beckley

John, I would like to try and do two things. First, I don't have your background to feel that compressive is irrelevant to the performance and I am willing to listen to that. Years ago we tried to follow the general philosophy of let's get a test that tells us a little bit about everything and we were strong on flex because we felt we were getting a measurement of the fiber's strength, resin fiber interaction in the mode, and then the failure generally turned out to be tensile on the bottom side but you were looking at compression and we were literally driven out of that comfortable data collection of flex by designers who said I don't use flex at all, I want compression. Various people to whom we listen essentially said okay, "we will collect a set of data in compression because the cost is too great to keep collecting both sets of data."

John Koenig

That leads to another question I'm going to ask. Everything that we have talked about so far is at the prepreg level and up. Is that the limits of our charter?

Bill Hall

No, that is the next item on the agenda. The next chart is the material supplier flow chart for the nozzle material today. Please check that for accuracy, it may or may not be completely correct. For Hitco add CCA3, the type of cloth that Hitco carbonizes. One thing, as John just mentioned, almost all the properties that NASA is interested in is from the prepregger's standpoint. We pick up incoming materials to the prepregger which

would be the carbon cloth, filler and resin and then we measure the prepreg properties coming out. This, of course, is what Thiokol does with it. If you look at the flow chart, there is a lot of materials processing to the left of prepregs that really influence prepreg properties. If you want to go one step back from AVTEX it would be that your pulp is supplied from Canada in white sheets. Do we take that for granted that the pulp is a constant? I am not sure that we should. I assume that most of you are familiar with the flow chart, where the carbon cloth starts off as rayon which is manufactured by AVTEX, who sends it out in spools to HIGHLANDS. HIGHLANDS weaves it into a 8 harness satin cloth. This is then shipped to all three carbonizers; POLYCARBON, AMOCO (is at Fostoria, Ohio, and HITCO) who carbonizes it. Two resins BORDEN (SC1008) and IRONSIDES (91LD) are qualified. One or two fillers, I don't know which, is used. However the filler is not on our specification list, but a specification for filler material is being prepared. Up till now there has been no information on the filler. The prepreppers take one of the cloths, resins, and fillers in almost any combination. Is that right Don?

Don Beckley

The modes of operation at the two prepreg houses differ in that way. The FM5055 from us is only one combination of materials, we would give it a different grade code or at least a suffix if we ran the other two fabrics.

Bill Hall

You are talking about HITCO cloth and IRONSIDE resin as your combination?

Don Beckley

As a single combination that is FM5055.

Bill Hall Although you do use the other cloth at other times. Is that not correct.

Don Beckley When specified, we will use the other cloth. We are qualified on all three.

Bill Hall FIBERITE goes essentially with POLYCARBON cloth and BORDEN resin, Is that correct Ed?

Ed Hemmelman We have used all three cloths.

Bill Hall Do you call it MX-4926?

Jon Weispfenning It is MX-4926.

Ed Hemmelman Morton Thiokol knows what fabric we use in each lot of prepreg.

Jon Weispfenning Over the last couple of years, it has been probably been about fifty-fifty POLYCARBON and HITCO.

Don Beckley One thing that prepreggers feel responsible for is the materials that they put into their product. There are rayon specs, fabric specs and there are filler specs that govern and essentially control those products to the same degree that has been found to be necessary for what has been delivered for the last twenty years.

Bill Hall These are specs for the rayon fibers or rayon cloth? Again that goes back, to our previous statement, you prepreggers are the one that qualifies suppliers, so our first interface is with the prepreggers.

Don Beckley The message at one time was that you were controlling down to the level you felt was necessary, and that was the prepreg level with the knowledge that someone was doing something below that.

John Koenig One last question. Are we specifically aimed at the SRM parts that are fabricated by Morton Thiokol because that may drive

what are the key properties or are we generalized to any NASA use of these materials?

Bill Hall

The original contract came out because of the problems with the nozzle material and MSFC started trying to find out why and they realized that Marshall did not have the expertise to really check into why we had this problem. So the Integrity program was started to bring Marshall up to a level where they had competent people that could converse with the industry people and know what was going on in the nozzle business. So it is a nozzle driven program.

John Koenig

Specifically, shuttle SRM.

Don Beckley

When the original Integrity program came out, there were some concern(s) about the space motors at the time to. So there was a slight slide over into carbon-carbon activities. John, I think that would now clarify that that would now no longer be part of the concern.

John Koenig

In thinking of the carbon-carbon, these same two materials are used on a good number of space motors and for that matter strategic launch.

Don Beckley

Out of our first Integrity contract, we did almost 50% on the carbon-carbon side material development. What you have done here is to say that that is no longer to be a following for this group.

John Koenig

If we consider SRM/IUS there might be different modes of response due to the design of that motor which would be something you would want to check for one could imagine. QC tests worth getting with some funny responses which would drive a tensily loaded structure which does exist in the shuttle SRM.

Bill Hall In agreeing with what Don said. The title of my first contract had carbon-carbon in it, but that interest fell, so that is now low priority. If you remember correctly, we had three failures of carbon-carbon nozzles about that time.

Bill Hall The next few charts have to do with tests that I know are routinely run and evaluated for the flow sheet that we just finished with. Please correct as you see fit. Starting with the cloth, the fibers are made by AVTEX who measure the following: ash content, sulfur content, zinc content, pH, finish extractable, twist, conditioned strength, conditioned elongation, moisture content, denier, shrinkage. Conditioned strength and elongation refers to either conditioned in a certain humidity and then test it or you dry it and test it. That is all the conditioned in front of those two items mean. Denier is a combination of a specific gravity and size. You weigh 9,000 meters of it and however many grams it weighs, that is the denier. A certain size, if it has different specific gravity, will give you a different denier. It is not a size only, it is a size-specific gravity relationship.

Bill Hall It is measured at AVTEX I think. I don't think it is done anywhere else as it is a fiber property.

Don Beckley It's just a big high number at that point.

Bill Hall You may not need to see it, right.

Group Mumble!!

Bill Hall There's a factor there for the ash. And then the rayon cloth, and I believe Highland does this, I think this is also done at the carbonizers, is that not correct, are these properties not measured there?

Jon Weispfenning They provide almost all of these for us and we also go back and double check, routinely.

Bill Hall Anything that we should add to that, anybody know of any other property that is routinely measured.

Don Beckley I think you could assign somebody to look over their specs as such Bill because I'm not sitting here with them I'm not sure that we have, uncovered everything.

Bill Hall My final report on this first three years grant will be out in August and they will be in there.

Don Beckley That's where you picked them up.

Thomas Paral We measure the ash content of the finished fabric and since we don't have a chance to look at the rayon yarn prior to use.

Ed Hemmelman What frequency do you measure them?

Thomas Paral In a forty roll shipment, we take about five rolls a rack and measure them.

Ed Hemmelman What do you do with that number.

Thomas Paral Historical value for us. In fact several years ago, that was one of the ways we solved the sodium problem coming up as change. It's a historical value for us, it's not required I don't believe in any of the specs. We do it on our own.

Bill Hall All of these properties are not specs, some of them are measured voluntarily and used for quality control data. I am not saying these are all in the specs. These are things that are routinely measured. I guess probably the carbonizer would be the only one that would have a feel for this. If there is anything else, that you feel should be measured, next time write me a letter and let me know.

John Koenig	Bill, have you documented all these specifications and will they be in your August report?
Ed Hemmelman	Is there any possibility of that section being distributed early.
Bill Hall	Yea, we can mail it out. You are talking about the test methods. We have copies of all those.
Ed Hemmelman	In the rayon cloth did we measure the denier?
??	I assume that they are talking about the fiber area.
Don Beckley	Areal weight up above it, tension, yes. It carries on if you are picking up a certain carry-over from AVTEX, but I think that's the only place that the measurement has been made,
Ed Hemmelman	Control of the extractable on the rayon cloth, when is that measured in? As received or after it goes through the washing process to remove them.
Thomas Paral	Primarily after the wash we do periodically check incoming raw material. Right now I am at a loss for how many times we do something like that. Primarily it is after the wash.
Ronald Higgins	Yes it's after the wash, Probably if we would find an excessive amount then we would go back and check it.
Thomas Paral	It's not a routine test to get an ?, normally after the wash. It has a tremendous affect on whether or not ... in the process.
Bill Hall	From the rayon cloth it goes through the carbonizing furnace. This is the carbon cloth properties which should be measured by both carbon cloth manufacturer and the prepregger. I'm not sure whether it's sodium now or whether you measure all of the alkalis and alkaline earths, does anybody know.
Group	It's just sodium.
Bill Hall	Just sodium, that's all?

Don Beckley That's a requirement. Each house may be making more whatever measurements they feel are appropriate.

Bill Hall We are still at 1200 ppm on sodium?

Group Yes.

Thomas Paral The specification actually has nothing in it, it is titled 1200 pm.

Don Beckley It has been removed from the specification. Which frankly made no sense to us as users.

Ed Hemmelman Is it appropriate for us to talk about the usefulness of some of this information?

Bill Hall Yes we are, remember the second part of our goal, if it has no bearing on the final use of it or the final properties of it, then I'm a firm believer in the fewer test methods and fewer specs we have, the better off we are. If we could just make one test and know that is was adequate that would suit me fine. I don't like tests just for testing.

Ed Hemmelman Going down the list from experience moisture content and thickness (Properties Measured on Rayon Precursor graph) I'm not so sure that those are data points that people run the tests to meet a required value and you better not test it again because it will all be different. To me thickness is just a function of areal weight and thread count.

Bill Hall I don't either. I don't have any idea, Anybody? I thought of that several times, why measure it. You've got the mass per square so, isn't that all you need?

Don Beckley I'll make a comment on moisture. In today's fabric, it is much less relevant than it was back in the earlier time for carbon fabrics, there seem to be a break point several years ago when sitting in the ambient conditions at Gardena, and that is really

what the moisture or the measurement was taken, then it was above six percent, all manner of problems accompanied the product, and now you can really better understand why and what happened of the prepregger. It really made a big differentiation between material of a 6% than stuff that was under 6% which effectively ended with a spec at about 3% as the margin we wanted to have on the property and what it really was dependent upon was the fact that the climate at Gardena was relatively stable and we weren't subjected to 99% relative humidity days you know to upset the number so that you could tell the really bad material from material that was "normal" and in that way if you ever got a six percent number again I would be very very concerned. It does its purpose. The whole question is should you continue in measuring something that 99 or 100% of the time is proven not to be a problem to prevent the one problem that might have happened, I guess you would have to imagine a roll that went through the processing turned black but had never really been processed at temperature to try and imagine that one bad culprit.

Jon Weispfenning

I think hidden in that requirement of the moisture of 3% is something that nobody pays that much attention on and that is preservation. I mean, how is that roll sealed, I mean that has to be more important to me the prepregger than whether that moisture content is 3% or not. It is going to suck it up. If I measure it today don't measure it tomorrow because it isn't going to be there.

Don Beckley A stable material and a stable climate doesn't suck it up. That is really the message I was trying to say from a previous experience.

Group Everyone talking at once.

Ed Hemmelman I think that we are a little bit at odds and there is somewhat of a concern about what Jon pointed out about preservation. I would like to make sure it is sealed in a moisture proof bag and would you not want to make sure that it was received like that by a prepregger.

Don Beckley That's not necessary. For normal material, it would be an extra cost to try and stop a stabilized material from going in and out it's normal conditions.

Ed Hemmelman Well, it seems to me then that we want to forget about moisture and come up with some other tests that will concentrate on a data point that would guarantee you that, it was processed the way you wanted.

?? The carbon fabric spec itself. It will launch a carbonizer into 2020/2150°F. Is that right?

Don Beckley Yes, 2200 to 2800°F.

Ed Hemmelman Or something like that. Isn't there like ten different materials within that carbonization temperature?

Group 7 or 8

Bill Hall Let me ask a question about thickness. Would this be a measure of the tightness of the weave, or not, of the original rayon? I don't know why it is in there. I am trying to figure out why it was there originally.

Thomas Paral I don't go back far enough in the history of the material to give me a good background, but from our standpoint there is no real

reason to be measuring that, the only thing I can think of is farther on down the road when the tapering the stuff. The thickness of the material then might have a

Ed Hemmelman Have you ever rejected a roll of carbon for out spec thickness?

Thomas Paral We have.

Don Beckley I believe it started from the standard textile specs all of which have thickness in them. As far as usefulness goes, over a periods there have been some people who have taken a real interest in thickness and tried to control it even tighter than it is possible to control, and they are looking at that factor of bulkiness in the yarn, in other words, how tight to all of the filaments collimate to each other. How much interfiber bonding is there and so on. There is probably some subtle things that thickness tells you, it's pretty *nubulous in the way of the technique itself is specified in the weight of the foot, size and weight of the foot, which really determines what number you get and you start off with the philosophy of; Do I want to see that bulkiness or do I want to push it-out of it and get down to a hard number so to speak, and we have kind of gone in between?

Ed Hemmelman If you see a role that is out of specification in thickness, what does that tell you about what happened in the process?

Thomas Paral At the point that we catch something like that or we see something like that, I'm not sure that we could identify specifically whether it was something we did or the weave, how much crimp they got into it or filament bubbles, there is many things that could affect it. I am not sure that we could identify that. We have many nice specifications to ship material. And not all specs are the same for thickness.

Ed Hemmelman	So, theoretically, that particular roll of carbon went to another program that has a different thickness, do you feel comfortable that you have shipped a good product?
Thomas Paral	From our standpoint, I do.
Ronald Higgins	There is a direct correlation between the thickness of the rayon cloth and then what happens to it nobody has ever thought of that but I think it probably could. When that starts to get near that lower edge of their tolerances, that's when we start having problems.
??	Is it not the same as areal weight?
Don Beckley	No I think what we're saying is that it is independent of it, to a degree.
John Koenig	Sometimes the weight will be up and the thickness will be down. So I wonder how tight the ...
Don Beckley	You have to look at both numbers at the same time to see it.
Group	Discussion
Ronald Higgins	When the thickness of the rayon cloth is thin then it is possible that when we carbonize it, it will also be thin. ... product we can't sell it is
John Koenig	Are we talking about a response behavior here?
Pat Pinoli	No, just the thickness itself. I would like to go back to previous discussions with regard to the moisture sensitivity of bare fabric. As part of my characterization work on fabric, I performed a lot of water adsorption studies which developed classical water vapor adsorption isotherms. The following plot shows the adsorption behavior of bare fabric to various relative humidity conditions. The time dependency at say 50% RH, which is common in California, is very pronounced. After 4 hours the

fabric will adsorb about 4 w%, however, after 96 hours adsorption has increased to over 8 w%.

Don Beckley

They are all at one temperature.

Pat Pinoli

Yes, all of the isotherms were developed at 23°C which best represents storage conditions. This behavior is classical for activated carbon and shows how sensitive bare STS/grade carbon fabric is to moisture adsorption. At 100% RH the fabric will saturate out at 14 to 15 w% water very quickly. Adsorption at 50% is much less within the times measured in this plot but note that substantial moisture continues to be adsorbed between 25 and 96 hours; week of exposure will result in adsorption significantly above 8 w%. Ref. Figures 1 and 2.

Don Beckley

Okay, but Pat historically would never measure more than 3% for a long long long time, your numbers are not what the real world is seeing, there what's happening in the laboratory. And the same thing, you move to Santa Ana and you measure it again and it is still under 3%, the seal is a polybag that is tucked in in the end, there is no sealing.

Pat Pinoli

As long as you don't have the flow of fresh 50% RH atmosphere into the bag it will dry out, what you are doing is creating a atmosphere depleted of water vapors. This atmosphere will remain static until more moisture is introduced. A second consideration is the isotherms are reversible, i.e., saturated bare fabric will lose moisture by exposure to say 20% RH. The issue laboratory data vs. Santa Ana environment does not detract from the relevancy of data generated under laboratory conditions. These isotherms are correct and developed under controlled laboratory conditions to establish the fundamental

behavior of this material. Referencing the Isotherm curves, your 3 w% experience suggests the fabric never sees an equivalent environment above 40% RH. Ref. Fig. 1.

Don Beckley Polyethylene actually breathes water so you know over a period of time that roll will equilibrate wherever the humidity is. At the WVTR of polyethylene is enough to depend on whether the fabric is a sink for moisture or you know trying to get rid of it.

Pat Pinoli The bottom line is what we've been going round and round for 10 years to establish. The only way to make a reproducible prepreg product is to predry the fabric before you go into the prepreg operation. Who cares what it has before you start your prepreg operations: 2%, 4%, 6%. Take the water out and then put it through your dip tank, now you've got consistent process. It is not important how much water you had in the fabric before prepregging. It cuts down on a lot of cost factors and it makes it a lot cheaper to ignore the bare fabric water pick-up. You indicated to me a long time ago that you do predry all of the materials. There is an indication that Fiberite does not precondition the bare fabric. This is a variable that I would like to see industry be consistent on.

Don Beckley We're not here to drive that one together.

Pat Pinoli I don't have any answer to render processing commonality, but I do feel the issue of residual moisture in the bare fabric is a valid concern.

Don Beckley I think while the subject is on we ought to make this point. You can predry it and it will come back and equilibrate after resin is on it at whatever it wanted to be before hand to a very great degree.

Pat Pinoli Ultimately, it has to see the atmosphere and the bare fabric, prepreg, preform and component will reflect the bare carbon fabric hydrophilic characteristics.

Don Beckley Well, it does if the prepregs it is going to be available to the atmosphere and it will equilibrate there from the time somebody starts to make tape out of it, it is going to be out in the atmosphere and essentially will equilibrate where it wants to. Fabrics that were of high moisture content and the classic one for me is Pluton that had an equilibrium moisture level of 12% or 15%, you could dry that thing to bone dryness and in 60 seconds after you finish drying it, it will pick the moisture back up with resin on top of it, so it is really no answer to do anything other than to make the fabric within a controlled level of its characteristics.

Pat Pinoli Dealing with activated carbon fabric in the STS/SRB components is a problem which starts with the bare fabric and continues through to the end product. Moisture control must be instituted through each step of the process to insure a consistent end product. Your opening statement indicated a desire to produce a consistent product; by ignoring moisture levels in the fabric during processing are you not deviating from that position?

Don Beckley No, I don't think so. But you said we know what the levels are.

Pat Pinoli I would like to address a technical point with regards to the active sites and pore structure of CCA3 fabric. My concern relates to drying out the bare fabric; removing water from the active sites and subsequently exposing the active sites to solvent in the 91LD resin. This will tend to increase the carbon fiber solvent adsorption. Is the end product better with a high

concentration of water or alcohol within the fiber? The sensitivity of CCA3 to adsorb IPA is much lower than water due to molecular size and polar attraction. These are the complex factors which will always be present when dealing with an activated carbon fabric.

Ed Hemmelman Well, aren't we kind of deviating from the charter? Now we are talking about getting into the process of manufacturing carbon cloth and not its quality. Should we be doing that at this committee meeting?

Bill Hall Only if someone feels like that has a direct effect upon the final properties. We are chartered to look at anything that would have an influence on the final properties of the fabricated nozzle by Thielkol. If you all feel like this is, you as the committee, you determine what we're going to look at. Okay, we'll move on and come back to this if the committee feels like it should.

Bill Hall As I say there are no specs on the filler at the present time, but these are the minimum recommended things that we look at. (See Figure Properties Measured on Filler)

Ed Hemmelman I think the spec as far as carbon assay is concerned is 90% minimum.

Jon Weispfenning Yea, indirectly the spec requires us to test 99% carbon black to see if meets 90% carbon present.

Don Beckley You have to realize that not all fillers over the history of the last 20 years have been 99%, that's the reason for the 90 number that was there at one point. I'm not saying it can't be updated, but it is a ... (everybody talking).

Jon Weispfenning Indirectly there are requirements to get that done.

Don Beckley I would like to register that I think one of the most important properties if not the most important is that the carbon assay be measured and maintained. So I think that's we've got to put carbon assay on this list.

Bill Hall Okay, add carbon assay, and we will follow up on that.

Thomas Parai Bill, what is TGA?

Bill Hall It is weight loss versus time and temperature.

?? You can buy varieties of carbon black that have half percent oil in it to keep dusting down. Maybe that's what they were looking for.

Bill Hall I'll check on the TGA and I'll let you know, that will be one of my items to check on.

Bill Hall Probably anything that comes off, moisture, oxidation, etc.

Don Beckley When you see this you can reflect that there was a period of time, let's say roughly two years ago, that NASA became very concerned and TGAs were run on everything. We run TGAs on fabric and TGAs on filler so I think that's where it came from. Whether it's totally relevant or not is open for discussion.

Pat Pinoli Data from TGA is complimentary to an ash content. The ash content measurement involves oxidation of any carbon and organic matter; leaving an inorganic ash residue. The ash content measurement involves oxidation of any carbon and organic matter; leaving an inorganic ash residue. The ash content test therefore treats both organic matter and carbon as oxidizable constituents. The TGA can provide an indication of organic volatility when run under inert atmosphere and in air can provide oxidation rate data. The thermal behavior of organic matter in the filler could be very important.

Bill Hall We'll check on the TGA.

Ed Hemmelman If that wasn't important why wouldn't they want to run TGA on carbon cloth. It is a major component compared to the filler.

Don Beckley I'm not certain, I think one of the current fabric specs has TGA in it for the fabricAbout every six or so roles you run a TGA. There's no criteria established and so it is just being collected as a data gathering at this point.

Bill Hall This is run on the carbon cloth. Then we need to go back to the carbon cloth tests and add TGA.

Don Beckley The trouble that we have observed on that test it is really sensitive to air flow and the amount of air, the size of the sample, it is virtually, well it is going to be very difficult to put a reasonable spec on it just because of the test method here respectively whether there is any variable in the fabric or not. We have two different TGA apparatuses and the apparatus themselves give different answers.

Bill Hall Two properties on this overhead are obviously misspelled; resin flaw is resin flow, and interlaminar is interlaminar. (See Figure Properties Measured on Prepreg)

Group Discussion

Bill Hall Let's briefly check the properties that are routinely checked on the resin and see if there are any additions, deletions, or comments on these. As I mentioned before, Chang's Index is a temperature measure of the amount of staging in the resin.

Ed Hemmelman That is only applicable to 91 LD, that test is not run on SC-1008.

Bill Hall 91 LD of course is the Ironsides resin used by U. S. Polymeric. The Infra-red base line and the GPC are two tests that are just

beginning to be used. Hopefully, we can get enough base line data on those two test so that they can become more usable.

Lou Ann Gibson is going to talk a little bit this afternoon probably on her work with GPC on the phenolic resin. Any comments on the properties on the resin? We can live with that.

John Koenig When you say we can live with that, is what is we are going through today in a sense of overview.

Bill Hall Right, you all come from different areas and a lot of you are very specific in your interest and hopefully you will go back and look at these. Those of you that are in this area may come up with suggestions during the intermediate six months period before we meet again.

John Koenig I just wanted to make sure that if a view graph goes out, it does not apply to people on the committee and I see a report says

Bill Hall No, this is just information to bring everybody up to essentially the same level. We started with the materials flow chart showing the complete materials process and this is just going through it again showing at each point what tests that are generally or routinely made.

Bill Hall Tack again is just a round about way of measuring the amount of staging, I have the spec, or I assume you know what they do, they take two sheets and lay it together with some overlap and press it and pull it.

Ed Hemmelman We haven't run that test.

Gary Hall Gel time and

Don Beckley It can be run as a pick gel right on the edge of the flow specimen. It is not usually run.

Ed Hemmelman Typically we control a gel time of heat resin and mixed resin that is where our control comes in. I have a problem with gel time of pick gel. Everyone in this room could run that test and get as many answers as we have people. It is really a subjective test.

Bill Hall Things like that are test we should look at.

Don Beckley It really happens if you are getting two dimensional heating some from the top platen and some from the bottom platen and so on and how far out are you so that it gets a degree of cooling. It is very difficult to stabilize the thermal environment that it is in. Fortunately and almost always the number looks very very similar to the initial resin gel doesn't hardly change a bit so therefore it has never been a really necessary thing to monitor, it is controlled somewhere else monitored somewhere else and we can get along without making dual measurements of the same property.

Ed Hemmelman You have to control the gel time and resin before you get to the prepreg. Because there is nothing I can do during the process to change it.

Bill Hall Any other comments on the prepreg list?

Don Beckley Well they're certainly the most important parameters to us as prepreggers, especially the uncured numbers. Cured numbers are like verifications. The climate that we are in for a desire for increased accuracy and so on, really suggest that we could use better methods than we have so I'd like to say that I think we are open but I don't know what they are.

Bill Hall On the press cured ones, I thought we had struck that one,

John Koenig It is out of the shuttle specs.

Bill Hall Are you all still running the test for you own information for your own information?

Group Yes

Bill Hall That was one of the few things that did change from the first go around was that the residual volatile test on the process cured material.

John Koenig We would be willing to have it put back in the specs, if some of the pretest conditioning accurately prepared the test specimen to generate useful data. Other than that I don't want to go into details.

Bill Hall We went that round, remember? I really wasn't quite sure as to what tests you had run. These were the only ones that were in the specs and that's all that I knew you run. You may run more, if so, please tell us so we can add them to the list. This is now tag-end type test. This is after it has gone through the real curing process and this is supposedly cured just like the nozzle itself. They run specific gravity, compressive strength, which Ben is going to talk about later on, resin content and residual volatiles. Residual volatiles are going to be discussed by both John and Pat.

Don Beckley But we are saying we think the current nozzle mode has no residual volatile, is that right?

Bill Hall Hardware is still there, we are talking about the cured prepreg.

Don Beckley Yes, I understand but I thought that was eliminated also. Is it?

Group Mumble

Don Beckley It's strange that they got it out of the prepreg specs.

Bill Hall No, the prepreg specs were made for press cured specimens.

Don Beckley Yes, but I'm saying that it is strange that Thiokol succeeded in getting it out of the prepreg spec where it never caused anybody any problem because the real issue was parts.

Bill Hall Right, but that is the way they got it out, they said that your having this trouble meeting it but yet it isn't cured the same way that the real part is cured. It really has no meaning compared to the final parts. That was one of the justifications for taking it out was pressing it between platens is not the same thing as curing it in the autoclave/hydroclave vacuum type environment that the nozzle is cured in.

John Koenig I think what they did is they extended the volatile test ...

Don Beckley That's for the prepreg. Yes, but I'm saying I think really that they don't measure RV on parts anymore. We will confirm that later on.

Bill Hall I've got about four more things that lead us into Pat's discussion and then we will take a break for coffee. One of my statements through the years has been my objection to saying you are going test fabric breaking strength under ASTM D1682 and stopping right there. There are eighteen variables in 1682. I have never seen a breaking strength reported other than X pounds per inch. They never give these numbers or letter numbers after that to indicate which type of test it was determined on. I think that it is not enough difference to worry about, but different tests and different testers do give you different results. And when you say you do it by D1682 that's not enough.

Gary Hall In the ASTM D1682 chart, what is your normal ... G-T, G-L?

Bill Hall

It's goes with the type of tester that you have at the top versus the way that you prepare your specimen on the left. Gary, are you familiar with that. You can take a sharp blade and cut out a one inch strip. You can cut out an inch and a half strip and strip-out the excess fibers and bring it down to an inch width. That's called a ravel test. If you just cut one inch it is just called a cut test and the grab test is where you have a wider strip and you have more narrow grips that are a specified width. And the grab will give you a higher value because you have the benefit of the textile on the sides helping share the load. The textile people like the grab, if I'm not mistaken. I have no problem with any of them, I'm just saying that you get different numbers depending on what method you use. All of these things are just pointing out all the different tests you are running. It should be 50 pounds GT or 50 pounds 2RT. Pat will go into more detail on that during his discussion. Back to residual volatiles, we conducted a round robin (MSFC, U.S. Polymeric, MTI, and Fiberite) on residual volatiles which is the test everybody said that we looked at before. This overhead shows the percent residual volatiles as tested at Marshall under the then current method for doing this test.(See Figure 9) The little cross marks are each test, there were four specimens, and you can see that the precision is wonderful, okay, no problem with precision. This was through 48 hours, this is the same test carried out to about 368 hours (See Figure 10), and you can see that after about 100 hours for this particular series, the percent residual vols leveled out and there wasn't much change from then on. This is at 325°F in an air circulating oven. Now

Pat, I have a couple of slides I've received from you. I carried it one step further as Pat had different size cubes. Both Pat and John mentioned, whenever you are measuring this, the really driving force is the difference in the concentration of your water or your volatile (whatever it is) inside the cube and that on the surface. If you don't remove the vols on and above the surface, you aren't going to get anymore out, this is the old drying phenomena and there is two ways to remove it: one is to have circulating air, and second is to do it by vacuum, but you have to get the vols away from the surface. Now to get them to the surface is another problem. It's a two prong phenomena, you have to vaporize it and then transport it from the interior to the surface. Then then you have to carry it away from the surface and you can do it by circulating air or vacuum. And the driving force basically is the difference in the concentration.

Pat Pinoli

The rate of volatile movement from inside the CCP to the surface is what I call "permeability". This desorption process appears to comply with Fick's law of diffusion which applies to absorption and not necessarily to adsorption.

Bill Hall

I'm an old ceramic man and if we talk about the moisture content of a silica brick, ten people could measure it and come up with the same answer because the silica has no affinity for the water, it is just there, and there is enough permeability for it to get to the surface. However, when you talk about carbon phenolic composite it's a different situation. The carbon cloth has a high affinity. The permeability isn't as uniform as in a silica brick, so it's much more difficult to determine the moisture

or volatile content. Most of it is water, I think, most of us will agree with that, that we are essentially talking about getting water out, there are other volatiles, light organics that come out that are relatively small. So when we start talking about the percent volatiles in a composite, we have to look at all of these other parameters. Some methods do not overcome the barriers, others do, and you are going to get different numbers. Which number are we interested in?

Ben Neighbors

I have one comment to make. This is a good example of changes we can make to improve the current specifications. When the specifications call out a specimen size of $(0.5 \pm 0.060) \times (0.5 \pm 0.06) \times (1.0 \pm 0.06)$ and you calculate the difference between the maximum and minimum volumes, there is a 54.7% difference in the mass of the material. So depending on the size of the specimen you can end up with almost any number for residual volatiles.

Bill Hall

This was a point Pat was making on this particular curve, he had three different size cube. The square was a 1/2 inch cube, the circle was a 1 inch cube and the triangle was a 1-1/2 inch cube and you can see the triangles lag significantly behind the other two. It is rate of diffusion and how far you have to go, see. Path of diffusion, distance. You just can't get it to the surface. Remember you have to get it to the surface and then you have to remove it once you get it to the surface.

Ben Neighbors

This is a very good example with the types of things you can do that are very simple dealing with tightening up on your tone and collect your data.

Bill Hall Because the the path to the surface was much longer If you have the maximum overtolerance in each direction. This is an interesting one compared to the other ones, you can see the difference in the curve. The time frames are the same. The temperature is the difference, now notice that. Diffusion is very temperature dependent and 220°F and 325°F are the two temperatures. This was at 325°F and this was at 220°F, now this is a vacuum, so he's removing, there is no problem removing it from the surface. He's is pulling it and sucking it right out. But, he's just not getting it to the surface. So, this points out again it is at least a two prong or three prong phenomena that we are looking at and it's not simple when you start talking about these preregs.

Pat Pinoli You can also see variations in permeability factor for any particular piece of hardware very difficult. The specimen size becomes a critical factor which must be determined to insure the perceived permeability value is relevant to hardware performance.

John Koenig There are two issues we are addressing here. One is how to run a moisture absorption test meaning the quantity of moisture. Second, is the complicating factor of the permeability of materials to the moisture which maybe a very critical monitor in the curing department.

Ed Hemmelman I would like to see evidence of the data.

Pat Pinoli I don't think that you are ever going to see all of the data in front of you to compare the performance of the material.

Ed Hemmelman If that is exactly true, I wonder if that will ever be available.

Pat Pinoli The last two years of study on the issue of residuals volatiles has developed analytical test methods which appear accurate and

reproducible. What is needed now is an extensive data base which tracks residual volatile levels through the processing operations and develops a permeability factor for the fabricated CCP.

Bill Hall I am going to put these together. The bottom three from the vacuum and the top three is the air circulating. You have to assume that these are going to get up to that level if not higher.

Ed Hemmelman The data will come together.

Don Beckley I think you have the driving difference of 325°F versus 220°F and I would be uncertain that they will ever get together just because of the thermal vibrations at 220°F can do no further curing of the resin system. It may not ever be as much as what happens with the 325°F. I wouldn't know that is going to happen Bill.

John Koenig If this is going to be a major issue, then it is something that we need to talk about.

Pat Pinoli I would like to talk about some of the issues that you get into with regards to Karl Fisher. Some of the advantages, disadvantages and the factors that have to be considered. In some ways, I decide in my own mind what I can justify and what I can't justify. I'll throw some of the subject out for discussion. Karl Fisher gives us more than just a number with regards to the water content of hardware. It tells you the percent of water that you have in the hardware with respect to a temperature. If you choose a temperature that is in the neighborhood of 325°F you find that the diffusion rate that the moisture coming out of your specimen is quite slow and so you end up with a time

dependency problem that you don't particularly like for laboratory operations, and then you have a test that is taking you four hours just to get one number, I don't like that. You want things that are relatively fast. If you increase the temperature in which you make the measurement to what I feel is a realistic number like 500°F the diffusion rate is quite high. You can get a very stable number out after two hours. In fact, a little bit of additional work would probably show that most of the specimens will desorb in about one hour. Now that makes it a realistic test because you can triplicate test within a four hour period and provide a reproducible number. The second Karl Fisher data of value is a total volatile content number and you might equate that to something that is fairly close to what you get with your existing test. With one exception, the Karl Fisher operates under an inert-atmosphere, and we've done enough work at Palo Alto to show the fabric is extremely sensitive to oxygen chemisorption. You have to think in the terms of a specimen that is left in an air atmosphere for 24 hours, 48 hours or longer extended periods as sitting there chemisorbing oxygen. Most people think carbon has a relatively low capacity for chemisorbing oxygen. But we've seen numbers like 1%, 2%, 4%, and 8% weight percent numbers of oxygen can chemisorb on activated carbon. Likewise, the phenolic resin itself may be sensitive to a certain amount of oxygen; my argument and the comment I would like to make is that if you are going to expose your test specimen to an atmosphere, it should be inert. Common sense dictates that we don't create an additional problem with regards to accuracy by exposing the sample to

oxygen. A third factor comes out of Karl Fisher is that you get a percent of the water in the vols. You can calculate very quickly how much of the total volatile content of the specimen is water. Typically, we run anywhere from 80% to 95% and what we can begin to see is some specimens having a higher concentration of CO_2 in the material and that can be an important number. A fourth factor that may be generated is permeability factor. We run 15 minute increments and as such we can plot out the diffusion rate of the vols coming out of the specimens. John and I have come to a pretty good agreement on data we need; Here are two factors that we would love to have data on. One is the total vol content of the material, and second, we want something in regards to permeability. Because pore pressure inside the hardware is controlled by these two factors. You can have an extremely high residual vol content but if it is permeable, you will never see a pore pressure build up. ... you can drill holes in the specimen and you can vent off the pore pressure or you can have microcracks which vent pore pressure. So we need a handle on permeability, something quick, accurate and reproducible. One of those data needs that everyone would like to have but find it difficult to get. I think that Karl Fisher can be employed to provide permeability data. You must be prepared to make pre-judgments like any test and set some standards. Like; what is the sample size you are going to use and what is the temperature you are going to use to measure the permeability. One of the things that John is going to be talking about is permeability with respect to moisture intrusion. The diffusion of moisture going into the CCP

specimens is very interesting data to develop and you need this data for environmental exposure. From the standpoint of hardware performance however, I'm more concerned about how fast will those residual vols come out of the hardware at launch. This could be a totally different permeability number with respect to what you get when moisture moves in thick CCP component. Now, I'll throw a few other factors out, Karl Fisher to me is has shown extremely good accurate reproducible results. It has taken us a year to work with the data and the technique to understand how to resolve such issues as background (blank) water content numbers. In other words, when you are running a test that is extremely sensitive to water, there is always a background water level in the sweep gas. Water is pervasive and a laboratory analytically test for water concentration level must be concerned about how much water is being brought into the apparatus by the sweep gas. You have to have an accurate number because perform differentials runs; In other words you run in increments, every 15 minutes we make a calculation. By this technique we have to subtract a background water value at each one of those increments. If, the background number is inaccurate by a very small value, we are multiplying that background number by 8 for 2 hours operation and this error can be significant. This is an issue that I have been looking at and feel confident that we will resolve.

The other questions you have to ask yourself is the size of the specimen you are working with. The 1/2 by 1/2 by 1 inch specimen is a substantial piece of material. It gives you what I would classify a macro data point. When you begin to look at

specimens that are 7mm by 7mm by 7mm, you have to ask yourself is that the best sample size to evaluate a piece of hardware. Are you going to make judgments on specimens that small by which you are going to accept or reject a big piece of hardware? Residual vols are extremely sensitive to the volume fraction of resin that is in the sample. The majority of the vols are trapped in the resin and the remainder is trapped in the fiber. We are talking about a CCP composite with 75% of the water in the resin and 25% in the fiber. If you were to go to say FM5014 with a WCA fabric then 100% of the residual volatiles are in the resin and none in the fiber. The TMA behavior of the composite TMA doesn't seem to care whether the water is in fiber or resin. So the Karl Fisher data residual volatile levels can be related to TMA response. I have a report just coming out in which we surveyed hardware by Karl Fisher technique and we duplicated results the current technique which uses a half by half by one inch specimen. The current test technique for residual volatile level appears to be influenced more by specimen permeability than residual volatile level. In fact, the data that I have generated suggests that the current test technique is the permeability test. It is really telling you how permeable that material is in four hours. It's not giving you an accurate measurement of total vol content. I would argue for a test that gives you a feel for both. Total content and permeability because I think they are interacted. I think these are the numbers I want to look at and we are going to have to use to assess performance. So, to summarize, I guess what I would recommend is a small program that tested material, possibly

panels rather than going to hardware using the current technique with inert atmosphere and higher temperature. At the same time running Karl Fisher and using the Karl Fisher as a technique to give you "the accurate number" for comparison with the improved STS technique. Then stand back and look at that data and try to make a judgment and it is going to be a judgment call as to which way you want to go as far as the final spec. As it is currently structured, the Karl Fisher test is the best technique for measuring residual vols. It is the most accurate and reproducible. I feel very comfortable with it. It has some downside risks since it does give you a micro versus a macro type of assessment therefore I recommend triplicate tests and report on average. I have seen enough of the Karl Fisher data to suggest that chances are that you aren't going to miss anything with regards to the current test and you are going to get a lot more information out of it. If we do recommend the Karl Fisher technique, there will be some time required to get everybody up to speed on how it operates, duplicate capabilities, training people and get into the sensitivities. I open it up to questioning. Karl Fisher would be an excellent tool to look at prepreg also. It offers you a technique to look at the prepreg with respect to residual volatile content and track it all the way through the process. You can go to the pre-form see how well you reduced the prepreg vol content and finally to the end product and see how the process has worked out. But that is a research tool at the present time. I don't think we are ready to go in and use it as a production tool. I'd rather keep it in a research stage at this point for a while. Ref. Figures 3 - 17.

Ronald Higgins In testing that you've done over the years I assume it's been years.

Pat Pinoli Yes, it has been a couple of years.

Ronald Higgins Has the testing just been on cured prepreg or has fiber, fabric, and has it been on resin, also.

Pat Pinoli I've done limited work on prepreg, bare fabric and neat resin. Primarily for background information. Normally it has been tag ends and panels to develop a totally new residual volatile data base. Comparing the Karl Fischer with the existing STS data base has confirmed that we have totally different data. This is where we think we are at in reality with you. Some of the data that Bill showed; typical shuttle data shows hardware vol content of 1.5%; where in reality we are dealing with vol content anywhere from 3.5 to 4% and in some locations I've seen it as high as 5%. The aft exit cone we performed was extremely important to show there are regions within hardware that you have extremely very high residual volatile contents. This survey developed accurate data right at that interface between the glass and the carbon which was extremely high. Ref. Figures 13 - 15.

Don Beckley Pat, it is supposed be higher there with the amount of A-stage resin that is painted on and not staged out.

Pat Pinoli I agree, but for the first time we see the numbers, and explain why the glass tends to delaminate at this interface. That has always been a concern to engineering. These things come back sometimes literally blown apart in that region. I thought initially it was because of the dissimilar expansions between the two materials but, now I believe the delamination is occurring because of high vol content right at the interface. This is the critical

type of data that NASA needs to assess the problem of glass delamination. You now have an effective tool to survey and analyze residual volatiles in hardware.

Don Beckley Is it a correct view that as you continue to cure and then you look at the Karl Fisher number it goes down? Is there a finite amount of water, that's what you are measuring, and as you cured harder and harder as in post-cure even, does the Karl Fisher number become less if you take a subsequent specimen?

Pat Pinoli Oh yes, in post-cure you drive off a lot of the water from the CCP. Most of the water was trapped within the composite however by operating at 500°F some additional condensation reaction releases water that would not be measured by a test operated at 310°F. Ref. Fig. 8, 16, and 17.

Don Beckley Continuing cure of the system.

Pat Pinoli My data suggest about 0.5 w% water is released by increasing the cure temperature from 310°F to 500°F in additional condensation cross-linkage. Ref. Fig. 16a.

Don Beckley Well even in the standard residual vol test you cure a specimen for 2 hours at 325, you then take that specimen and in a residual vol test go 4 hours at 325, you see our classic number which is roughly 1.5 to 2% so just the 2 hours of additional cure at the same temperature are producing further water and as I perceive it if you go to 500° you've upped to delta and therefore you are going to see a higher number.

Pat Pinoli Yes, the important thing to remember is if you extend the time at 325°F you will double the volatile value and performing a Karl Fischer assessment at 500°F will give an even higher value.

Don Beckley But they are part of the cure reaction.

Pat Pinoli But it is all potential water in the composite.

Don Beckley But it's useful ablative water at this point.

Pat Pinoli Well, then you are getting into whether there is any useful role for the water in the composite, that's a fundamental question. We could be here all day.

Don Beckley One thing that I heard that is kind of new to me, you have identified the amount of water proportioned to the fabric in a carbon material versus the WCA material. Our data taken as an aggregate residual vols of carbon phenolics versus graphite phenolics has the same value on the same resin system with the same amount of resin.

Pat Pinoli Do you remember the curves that the bar charts I showed in the last meeting which we were discussing, in which I surveyed by volume fraction the amount of water in a carbon phenolic and as you transcend it into the glass phenolic region of the aft exit cone. You will see there was about 25% more of the water content (by volume) in the carbon phenolic versus the glass phenolic. The volume fractions of matrix vs. fiber in your prepregs are pretty comparable using your 26% or 36% weight percent numbers, and the only way you can account for the additional water in the carbon phenolic is to attribute it to the fabric adsorption. Ref. Fig. 13-15.

Don Beckley Yes, but now I'm going to give you another set of numbers and say I get 2.25% vol and graphite phenolic and 2.25% vol in carbon phenolic, the same cure, cured side by side, in the same press, there is no overall difference.

Pat Pinoli What you are saying is that there is no standard for measuring residual volatiles in CCP.

Don Beckley What I am saying is that the numbers don't differ.

Pat Pinoli If you are going to make a survey like you are talking about, you have to make sure both of those specimens represent the same cure cycle, fiber/resin volume fraction, some resin staging etc. all these factors and the test technique for measuring residual volatiles level are suspect.

Don Beckley In the same press at the same time.

Pat Pinoli We also have excellent data generated on NASA/M & N billets which confirm the fabric adsorption rule.

Don Beckley Everything is a constant in the numbers 15,000 measurement.

Both "Talking at once"

Pat Pinoli That is why we developed the aft exit data base which is excellent from that standpoint. The same piece of hardware is surveyed in many locations and determining the carbon phenolic has 25% more water than GCP. Ref. Fig. 14-15.

Pat Pinoli You have to also be careful because volume fraction of resin and not the weight fractions.

Don Beckley They are really about the same on those two products.

Eric Stokes That's not surprising when we are talking about the resin generating the moisture to start with.

Don Beckley But Pat's contention is he believes he is seeing a fiber component.

Eric Stokes Well I think that is because the moisture goes from the matrix into the fiber.

Don Beckley Yes, okay, but we don't see it, that's what I'm saying that we can't find the difference when I run graphite phenolic and measure residual vols.

Pat Pinoli By his method and that's part of the problem. He is using a test technique that doesn't have the accuracy of the Karl Fisher and can be influenced by oxidation and permeability factors.

Eric Stokes I still don't see any problem. You still have the water there, one is in the matrix and the other is partially in the fiber.

Don Beckley The question is, is it in the fiber or not.

Eric Stokes You have distinguished that from your technique, you've used the total moisture content for the two materials. You haven't said whether it is in the fiber or matrix. So that is no discrepancy between your data.

Pat Pinoli I think the point that he is making is that he doesn't really care if it is in the fiber or not?

Eric Stokes No, I am not saying that at all. I just didn't see where the argument was coming from. Obviously the water is being generated by the matrix and the resin material itself when it's being cured. You've got the same amount of resin there and it's curing the same amount, you have the same amount of water. Pat was just saying that part of that water goes into the fiber when you talk about carbon fiber.

Pat Pinoli It's activated carbon. How can you rationalize that it won't go into the fiber, you've got an activated carbon and you know it is sensitive to water, you know water is being released for the condensation reaction, where is it going to go? It is going to be trapped in the matrix and/or some it is going to migrate to the fiber. It is a diffusion problem. It going to want to migrate out of the matrix into the fiber and over any time period you can probably show that it is jumping back and forth. In fact I would go so far as to say that it wants to be driven to the

activated carbon. It would love to move out of the matrix at a constant diffusion rate into the fiber. That's getting off of the point. The point is that I don't think our charter is to look at the issue of activated carbon or non-activated carbon. It's to learn to live with what we've got. I've accepted that fact. We are going to have to live with activated carbon. From a standpoint of job security Don, I love it. I've got a job for life.

Group

Everyone talking.

Ed Hemmelman

You've got another aspect of the problem is that you know the phenolic resin during cure is going to release a lot of water. You've got to live with that or else we have to change our matrix and I don't think anyone wants to do that.

Pat Pinoli

That's what ASRM is all about. I don't think our charter is to look alternate resin systems.

Bill Hall

I would like to add one more test to the two that you suggested. I'd like to see the normal test temperature 325 versus both elevated temperatures. I think John indicated you would not get the same results. Just like they see it. When you run the sample, the air circulating and elevated temperature and inert, then do it in circulating air at 325 what we've been doing for years and compare. If you are getting the same curve and all you are doing is raising your number you haven't accomplished anything.

Pat Pinoli

My data suggests these changes will create a new data base, not just a standpoint offset.

Bill Hall

If you've got three curves and they are just offset you are getting the same results just a different number. I think John

frowned at me when I said that. He doesn't expect that, I don't believe.

John Koenig

I think there are a number of issues and Pat is far more experienced in the Karl Fisher test than us we expect a new test, but there are a couple of things that need to be considered: (1) If indeed, from the point of view of the volume of moisture and the measure of the quantity of the moisture, we are dealing with a much smaller specimen and then the test is more susceptible to the environment or perhaps requiring tighter controls on the environment that the specimen is exposed to. In a larger specimen we are able to maintain what is roughly a normal response within the material and we can go back and predict the response even further up to the size of SRM components where you get up to numbers of years before you dramatically shift the moisture that is at the center point, hence that number does have a realism to it. There is a moisture content. On a four inch component it is not as sensitive to the environment and therefore it is going to still be somewhat real at the time of flight. Taking this to the other extreme, when we run TGA we grind the stuff into a powder and the moisture measurement is almost a direct correspondence to the conditions in the room at the point in time of the test because the time it takes for the powder to reach the equilibrium is no longer measured in years or days or hours but now almost seconds.

Pat Pinoli

Like grinding up a watermelon. Grinding exposes more surface area to the environment and the particles can desorb or absorb from the atmosphere very rapidly.

John Koenig I think the second thing is something we started to investigate is if we are going to use the Karl Fisher technique (and we got our data and Pat has his ...), but when we take the material to 500°F, we are changing that material. And if one of the data points we are attempting to get out of it is the diffusion rate, we are changing the diffusion rate by more than just the effect of temperature on the diffusion rate. We are changing by the temperature we are taking it to changing the material. So while 500° is a wonderful temperature for maximizing the measurement or getting a measurement of all of the vols that are going to be sitting there, it will act on the material in subsequent events and may be a poor choice in terms of the measurement of the diffusion.

Pat Pinoli What you are saying, "Is it the magical number that we want to use for measuring the permeability?"

John Koenig Size and temperature are question marks on the technique, not against the Karl Fisher specifically. There is work to be done in terms of what we are going to obtain from the data.

Ed Hemmelman Wouldn't you hope to obtain from the data that you either fabricated a good part or a bad part, whatever a good part or bad part means.

Ronald Higgins That's a big controversy that is going on still, is whether or not the basic question of whether or not excess vols are bad or good. That really hasn't been decided yet. There are some opinions but no one said there is any hard and fast evidence one way or the other. What do you think Pat, you are involved in the design.

Pat Doan Like you say there are a lot of opinions. Nobody has convinced the majority yet.

Ronald Higgins That's a basic question that has to be addressed before you want to go.

Pat Pinoli I'd like to clarify that. Are we really saying that there is a consensus at Morton Thiokol Wasatch that high, extremely high, residual vols doesn't present a component performance problem.

Ben Neighbors I don't agree with that. You know what we historically ...

Pat Pinoli I don't want to get into designs, I know the road you are coming down. What I'm saying is given equivalencies ..., do you prefer to have low residual vols or high residual vols on a one to one system.

Pat Doan To my knowledge there haven't been any correlations between vols and performance.

Pat Pinoli The argument we have on that issue is on what basis do you judge residual volatile content. Your data base? Your current data base isn't worth a damn. Everybody recognizes that you are measuring permeability.... So how can you stand up and say residual vols don't mean anything.

Ben Neighbors You want what you been having, now the fact that we don't have a number that represents consistently the amount of volatiles we've had is another issue. We have to gain the data base to see what our total vol content is and ...

Pat Pinoli What you are saying is that you want more evidence, well that's what the TMA data has been doing. That's what restrained thermal growth has been doing. It has been looking at the role of residual vols in a composite. It show the dynamic affect of residual vols.

Ben Neighbors

We know that the hardware we have now is performing very nicely and we want the performance to continue, now as far as what the exact residual volatile content is of our hardware we don't know. Well, we have some indication from the work we've done and I guess my personal opinion is we want to stay where we are. The things that we are trying to do to our specifications is detect if something drastically changed. We want to have a test in place that says, hey your volatiles have quadrupled in value or they have gone down 90%. We are looking for differences.

Pat Pinoli

I don't think we have any disagreement on that.

Ed Hemmelman

Has anybody taken a look at the cure cycle and figured out if they got a high residual volatile number where it could have happened in the cycle. As I recall a couple of years ago, there were people who were looking at the shuttle cure cycle and recommending a long hold time at a low temperature. As I recall the results of that was no matter what they did with the cure cycle they couldn't affect a change in the residual volatile content. A test to me is only as useful as the discrimination that it provides. I am speaking as a casual observer here, because we don't make hardware. But if I did I'd like to have a test that tells me if I've got part, that it has seen the proper cure cycle, and I am confident that it will perform like the design said it should. My question is; we've got four tests listed here, do they do that for us? Is there some other test that might give you a better number?

Ben Neighbors

Which four tests were you referring to. Okay, this is what I'm giving my presentation on.

Pat Doan Is that not the objective here, of this committee. To find a test that tells you that's predictable and reproducible and then over here we have got materials and performance required. Aren't you guys trying to come up with a test to do what you are saying. None of these tests do to my knowledge, again I'm not the expert but none of these tests we do tells us how the nozzle will perform

Ed Hemmelman From a materials supplier standpoint, there is only a couple of things we could do that influences any given test on cured parts. I think the only single test that we have been doing is resin content. Either we make it to spec or we don't. Prepreg and resin content is going to affect resin content on cured parts. Specific gravity is a compositional matter.

Don Beckley Also, fabrication and along that line.

Ronald Higgins As a matter of fact, our section probably feels that most of the specifications we have in place were really driven more from not the end users, us, but because they were things you as prepreggers could judge to measure how good your process was. How you can apply the resin, and not all of the specs, a lot of them like resin content, vols, density, a lot of those, they don't tell us how the ... performs by looking at them until after you fire them over. I think that is the point Pat is making.

Don Beckley But you are used to a nice narrow band of those numbers, if you take any one of those and drive it...

Group How narrow?

Don Beckley Narrow compared to what it could be. In other words, the numbers that are set in there will give you a performance level.

Pat Doan No, I don't think so. This says predictable and reproducible and over here we have performance. Well reproducible is fine, you've can test anything and have your number the same way over again okay it is reproducible. Will that tell you how it performs.

Don Beckley I think the answer is that you have already set a range that works. Move well outside that range and you will find that it won't work. Take any one of those numbers and move it far enough out and it will give you bad performance. You are used to looking at a range and accepting the performance level that goes with it. Find yourself a specific gravity that significantly off and find a resin content that is significantly off and make it be a real number and you won't receive the performance that you are taking for granted.

Lou Ann Fikes I've got that data and I will present that in my presentation.

Don Beckley Well, you have done it before in your subscale motor tests. In the initial starting of the shuttle program we fired higher resin contents to give you a cheaper material, there is a very sharp threshold just a few percent up the road from where you are. You never see that because you don't get high resin contents as such and likewise get your specific gravity down below it. It is indicative of insufficient quality either due to material or manufacturing and it probably won't perform. You will see excessive erosion. I think we have experienced those before. I'll be glad to look at your data though.

Bill Hall Okay, is there any more about the Karl Fisher or the residual vols?

Eric Stokes I have one concerning the Karl Fisher and that is I don't think from the apparatus that we have been using that you can get a

single size as big as representative of the part. And the other thing is I don't know whether it is in the specs now, I'm not familiar with the specs, but orientation of the piece that you take out of the part is critical.

Pat Pinoli The current test procedure utilizes a 7mm cube, therefore, ply orientation is not critical. The other issue is diffusion time and that is set at 2 hours. This time is adequate at 500°F to achieve (essentially) total residual volatile desorption.

Don Beckley Comment on that $7 \times 7 \times 7 \text{ (cm}^3\text{)}$ It would lead you to be a difference of 1/12 or 8% depending on which phase of the fiber resin fiber relationship you are cutting into. You could either have one more or one less fiber..

Pat Pinoli Is your question "How many plies in a 7 mm cube?" If we assume a ply thickness of 16 mils, then each cube will represent 17-18 plies.

Don Beckley Well even in the other dimension. In the planar direction you've got about 48 yarns per inch and 7mm is somewhat like a quarter of that or a third of it so you have around twelve yarns that you are looking at, you can be on the outside of the twelfth yarn or on the inside of this one but move over here and you will be in the middle of it or you will be in the center, I just don't think you have enough specimen to give you a representative of the material.

Pat Pinoli I interpret your question to read: "Is the specimen large enough to prevent a random variation in volume fraction to matrix vs. fiber, i.e., if the specimen is extremely small, the residual volatile level variability could be primarily associated with fiber/matrix volume fraction variation. This concern is one reason for the

current 7mm cube size. Previously, we did a lot of analysis with 7 x 7 x 1mm ply thickness specimens. One mm only provides 2 to 3 plies; therefore, I was concerned if measured residual volatile level variability within a part was true indication or a reflection of specimen matrix/fiber volume fraction variance. If we assume your observation is correct and no water resides in the carbon fiber, the fiber/matrix volume factor is much higher than if my data implication is correct. With regard to fiber/matrix volume fraction variability, I feel the 7mm cube is large enough to insure minimal volume fraction variability. Your question may imply another question: "Is a 7mm cube large enough to effectively assess a large component residual volatile level?" A much larger, 1/2" x 1/2" x 1", specimen is currently being employed. This issue requires some understanding for the importance of very localized volatile concentration levels, such as 7mm cube size regions; which have higher than normal residual volatile levels. Personally, I prefer data which reflects localized region variability and triplicate 7mm cube specimen, average values, should be complementary to a Karl Fischer data generated on 1/2" x 1/2" x 1" specimens. Keep in mind, 3 triplicate Karl Fischer runs are cheaper, faster and more accurate than data generated by the current technique.

Eric Stokes

My concern is if you take that 7 x 7 x 7 out of a full part, by the time you machined it and taken it to the test facility, it has been seven hours at a minimum and who knows how much longer and this has a strong effect on the volatile content.

Pat Pinoli

Let's address that issue a little bit. We typically work with blocks of material which are usually about 3 to 4 inches thick

and before we extract the Karl Fisher specimen we cut it right down the center, and work out of the center of the material. I don't think we have to concern ourselves that residual volatile levels change dramatically from the time it was manufactured and when testing is performed. As long as we have been prudent along the way and haven't exposed the tag end to a rainstorm or stored it in a dessicator prior to analysis. From the minute a specimen is extracted; its weight is recorded and again weighed prior to test. So if there is any dramatic change in the specimen weight we have the data. Frankly, with the little bit of care we have taken, variability due to environmental exposure has been minimal. I haven't been seeing any dramatic changes. Some of the specimens tend to pick up a little bit of weight and some tend to comperably lose. I'm more concerned about dusting that can occur when the specimen is transformed into a ply bag than environmental exposure. Our current procedure is to test within 24 hours after the specimen is cut and the poly bag appears adequate to prevent significant moisture adsorption or desorption.

Bill Hall

Let me ask an economic question. Wouldn't it be much more expensive to machine those than what we are doing now?

Pat Pinoli

No, I don't think so. We rough cut our 7mm cube specimens and the current technique requires precision machinery. The question is: "Do tag end specimens represent the hardware?" My Karl Fischer data indicates yes and no, depending on how the tag end specimen was processed and extracted. Data generated on Aft Exit Cone S/N 6 and 12, was very extensive and showed considerable variability (+20%) within the part itself. Tag end

data on involute OBR components appeared very representative of the hardware; since the tag end ring was extracted from the billet ring middle section. I do believe that tag end segments which are processed independent of the component will reflect an accurate residual volatile lead.

Ben Neighbors

The ... data looks pretty bad. Maybe we better let you (Pat) give your data for that. We will be through my presentation here in a minute.

Pat Pinoli

I did throw the ... out. Some specimens and some tag ends are extracted from regions that are highly debatable.

Bill Hall

Okay, let's move on to the GPC. The purpose for this presentation is that we need to get a better handle on the staging of a resin at various steps along the way and we really don't have a good technique right now, and I'm not sure GPC is it. But it is something that, may like the Karl Fisher thing, it may be something for us to look at anyway. In the past several weeks, MSFC has had some work being done on glass phenolic. It started with Morton Thiokol and Fiberite and Marshall got involved in it and Lou Ann, an engineer at Marshall, did some work on it and she agreed to tell us a little bit about what they found out about that particular study.

Lou Ann Fikes

Okay, on mine I was going to talk about the gel permeation chromatography that is used in a project I was working on. I am receiving material from carbon cloth phenolic prepreg that goes into DM9, QM6, 7, and 8 in the first four flights. And this is Fiberite or U.S. Poly coming from Morton Thiokol. And we are doing a standard test on the prepreg as well as the extra test that we've added in like the gel permeation chromatography, the

IRPI, TGA, DSC, the list goes on and on. This is to gather data and see what we can do to characterize the material that is going into these nozzles. We did some work with those gel permeation chromatography, and you have to prepare your sample, do an ultrasonic acetone extraction, to prepare your sample to use for the GPC. You take that sample and you will do a roto evaporation which you will take the acetone out of it and you are left with your resin sample. Then you add THF, tetrahydrofuran, to it because that is what the system uses, it's a water system that the GPC is run through. You have a standard that you run and then you run your sample and it's normally two specimens per sample number. All that we can get from this test is molecular weight. And that I can use to compare one lot to another lot, and if you see some kind of variations in it I can also use an IR test to make some comparison. Now, I brought some data, and I brought an example of the type test that you get, the type results that you get with the test, you've got on the back sheet an example of the standard that is run. That standard, the retention times from that standard is used when you run your actual sample, you've got two samples that you run and then you do the analysis and from the analysis I've got underlined here weight average. That's molecular weight. The top sheet, this is going to be one shipment that we received from Thiokol that we did the GPC analysis and it's got molecular weight listed. Sometimes you will see two molecular weights listed out there, if we ran two different tests and the results do not fall right on top of each other. I'll pass that around letting you see the type of results

you get with the GPC. Now, we also did some tests on glass phenolic, and this was to compare the IRPI, the results you can get with this infra-red polymerization index, to get the degree of advancement and compare it with molecular weight. And there is one lot that really stood out in which we got some high values from the molecular weight. And then I compared with IRPI and you also got high values for that. So you've got some advancement that is taking place there. I've got a copy of the procedures for the GPC and the IRPI if you want copies of that to look at. But this is just data gathering for right now so we can compare one lot to another lot and like it has been said before you will get variations within that one lot just by where the sample is taken.

Don Beckley

Lou Ann, what is the roto-evaporator technique. What condition does it put the material under.

Lou Ann Fikes

It's heated and it is spun. It's glass. It's a heated water bath at 30°C. Your sample is divided into three parts and you roto-evaporating those three parts and your final resin is collected off of that.

Ronald Higgins

How long have you been using ...?

Lou Ann Fikes

Each part takes about ten minutes to roto-evaporate.

Ed Hemmelman

When do the extraction with acetone, do you take some precautions and insure that you have the extracted the total population of the resin off the prepreg?

Jon Welspfenning

Why do you use acetone rather than THF.

Lou Ann Fikes

The resin solids, the filler and cloth content, are all taken from this ultrasonic acetone extraction.

?

Using the same sample for the resin content test.

Pat Pinoli Is that a conventional acetone soxlet extraction technique?

Lou Ann Fikes Yes, you've got I have the procedure here if you want to look at that.

Pat Pinoli I'm just curious about soxlet extraction technique that involves some temperature. Are you advancing that resin?

Don Beckley The conventional ... doesn't Pat. The acetone is exposed to a 40°F kind of water and then dropped into that It is only if you let the resin go down in the bottom of the flask would it ever see any heat at all.

Pat Pinoli How long the extraction process takes is another factor.

Group Discussion

Ed Hemmelman In the resin content test you are boiling the acetone resin mixture. She is using an ultrasonic ambient temperature.

Lou Ann Fikes ultrasonic Acetone Extraction.

Bill Hall We could have some copies of that made if some of you want to carry it with you.

Pat Pinoli I am also curious about you seeing differences in molecular weight as you survey the prepreg. Doesn't that surprise you a little bit? Shouldn't it be fairly consistent.

Lou Ann Fikes Are you talking about from lot to lot or within a lot.

Pat Pinoli Within a lot. I would expect one lot to be pretty consistent. And when you said you saw some inconsistencies, I assumed within lot variability. Lot to lot variability should be expected.

Bill Hall This resin is coming out of a prepreg now.

Lou Ann Fikes And my samples were taken from three locations on the prepreg.

Pat Pinoli You are surveying across the width; salvage edge to salvage edge?

Lou Ann Fikes You will see some slight variations across there.

Don Beckley How reproducible is one specimen to another of the same sample.

Lou Ann Fikes Well, okay, there are two runs made on every sample and you can lay one graph right on top of the other. If not then there is something wrong with that sample. You prepare it again and start over.

Don Beckley So they repeat each other and the calculation turns out to be how different they are or do they stay the same? Between two samples are they always exactly the same number, say 30/40 difference in there. But this molecular weight you get is not the exact molecular weight of your sample. It's only a way that we can compare one lot to another lot. One sample from a lot comparing it to one sample in another lot.

Pat Pinoli It's a reference number, is what you are saying..

Lou Ann Fikes Right, I can't say that it is the molecular weight.

Don Beckley But in difference you are not trying to say that it is not number average or weight average.

Lou Ann Fikes I have it compared to my standard.

Scott Jackels What is your standard?

Don Beckley But your standard is a given number, and so presumably every number is so far to the...

Lou Ann Fikes But my number I can't say is the molecular weight. I just have to say that it is compared to the standard.

John Koenig How did your data compare with the infra-red type.

Lou Ann Fikes You will see that in the glass phenolic, the sample in the data that is coming around, you will see that there was a very high value for the molecular weight on a GPC and you also got another high value on the IRPI. So IRPI gives me degrees of

advancement whereas GPC is giving me a way I can compare molecular weight.

Don Beckley

In a normal IRPI though you will run into the point where you get so advanced and then you can't extract what effectively is more advanced, so you almost start to see lower numbers because you aren't getting the whole sample in the cell. So it is applicable only up to a certain point and then it isn't going any further.

?

GPC would be the same way with the acetone extraction.

Don Beckley

If it can't, if it is incapable of getting it out.

Lou Ann Fikes

So all of this is doing is just gathering data to see what this test would provide for us to see if that is a route we need to go. We are doing the standard test but we are also doing more tests to see what it is going to give us and that way we can go back look at the vendor certification data and compare it. Because, like the material that I receive, it's not tested before it's going into a part.

Don Beckley

Ultimately those two numbers are probably going to be worthwhile beating against a vol number and a flow number, and a resin number all put together to see if you can identify some reason that this number is more useful than those other numbers. Then we will be at a point of making progress.

Ed Hemmelman

Are you seeing any new trends or can you make any speculations regarding the data?

Lou Ann Fikes

Another study that we did involves an out of storage study where we took samples and had them stored out of the normal storage conditions of the 40 degrees, and left some that were not packaged in any bagging material. Then we had some that

were packaged in the aluminum bag and then some that were package in polyethylene bags. We looked at tests on day zero, day fifteen, and day thirty comparing the different type bagging conditions. I've gotten results like volatile content, we ran that. We ran resin flow, we did ultrasonic acetone extraction, and we also did the GPC. On the GPC we got results like on day zero unbagged an average of like 1178. On day fifteen it was 1377. Day thirty was 1329. Then take the same type sample, put it in a ziploc bag, the polyethylene bag, on day 15 tested it was 1318 compared to 1377 on unbagged. Then leave it for 30 days it's 1385. You've gone from 1318 to 1385. I'll pass this around and you can see it. Then if you take it and put it in an aluminum polyethylene bag you got 1349 for day 15 and 1316 for day 30. So this is another study that we did just to see what gel permeation chromatography could tell us. By leaving the sample out for thirty days either out completely with no bag with aluminum polyethylene bag or just a polyethylene bag you can see the results there and I'll pass that around.

Ed Hemmelman

I wouldn't expect that the material would change. In my way of thinking, molecular weight is a function of time at temperature. All things being equal I wouldn't expect to see much of a difference depending on how it was bagged. Of course, I am surprised by a lot of things in life but I wouldn't think there would be that much of a difference.

Lou Ann Fikes

We didn't know what to expect. Also there is not a desiccant included in this bag. It's just the bagging conditions. Seeing what the bags will tell us. The results are really surprising to us also.

Don Beckley What did you decide?

Lou Ann Fikes Well, we've gone back to do another study in which we are looking at the bag and conditions itself. We are taking a way that we can measure with a desiccant inside. I'm trying to think of a name I can use to describe the way we can measure the humidity and the temperature inside of the bag and take this instrument and put it inside the polyethylene bag versus the aluminum polyethylene bag and then put it inside the cooler by itself and compare that. So that's going on right now, a sixty day study to see what is happening inside of those bags either with a desiccant or without a desiccant. We are going to see what type bag is the best to use.

Ronald Higgins So the first study of the bags you didn't know the temperature. You didn't record the temperature ...

Lou Ann Fikes No, we have that, that was just in a room it was not in a cooler. We just took the samples out to see what would happen after 30 days leaving carbon cloth phenolic out for 30 days. Outside of the normal values conditions, outside the cooler we had a means that we could measure the temperature and humidity.

Ronald Higgins ...In the bag, the temperature in the bag ...

Lou Ann Fikes The bags are just placed in a room.

Ronald Higgins No the next study.

Lou Ann Fikes The next study we are doing involves putting the bags inside a cooler and then taking an instrument to measure the temperature and humidity inside that bag, inside the polyethylene bag and inside the aluminum polyethylene bag, and just set the instrument inside the cooler itself and see what variations we have.

Pat Pinoli Would you not also be interested in seeing what happens as you expose the higher RH? Will the moisture tend to reverse the phenolic resin reaction?

Ed Hemmelman You are violating some law of thermodynamics. It seems to me that it would be an irreversible reaction at least with my experience.

Group Discussion

Ronald Higgins Have you done this testing on the resin or on the neat resin?

Lou Ann Fikes No not on the neat resin. My project only involves the carbon cloth phenolic prepreg going into the motors DM9, QM678, and first flight. So that's the material that I am using.

Ben Neighbors First of all we'll start looking at the GPC on the ...

Lou Ann Fikes We didn't know what to expect from this particular test, and it's just showing here that on day zero you've got that molecular weight but once you've cut your sample with time we aren't sure what all it is picking up if it is moisture or what. So this just point out the understudies that need to be done.

Pat Doan So you are going up this You are not trying to make a relation between the bags?

Lou Ann Fikes Well we went back to do a study on the bags and that's what is going on right now.

Ben Neighbors What I've got up there (overhead) is just the top graph is the straight as the SE1008 mixed with the air The bottom one is a little different column sets so that the curves look a little different but essentially the same thing. The bottom is a typical prepreg value.

Don Beckley It is sort of relative though to talking about molecular weights and it is still really a small low molecular weight compared to most

other polymer worlds. We are really talking about going from 100's over here to what up here, maybe 10,000 on the top side. A lot of polymers are multi-million molecular weight polymers so this is really a narrow fraction. It has always amazed me that what we spend so much time talking about B-staging is really such a small molecular weight change. This is not very much movement.

Ed Hemmelman

What happens in B-stage, you get rid of a surplus amount of free phenol that is present in the resin.

Don Beckley

You can see that as a factor.

Group

Discussion

Bill Hall

Those of you that want part of this data that Lou Ann brought, if you will see her during the lunch hour so we will know how many copies of each to prepare. We can't ask too much of Fiberite but we will try run off everybody a copy that wants it or Lou Ann can take your name and address and mail it to you. It would probably be better to take it back with you so let's just find out how many we need and we will let her know at lunch.

Ben Neighbors

Currently this is what we are testing on re-designed motor tag-ends. First, we test residual vol and our specification limits are 0 to 3 percent for the carbon. And 0 to 3-1/4 percent on glass and 0 to 3 1/4 percent for the silica. The historical data is 1.8 percent and I've got an X-bar chart to show you what types of variations that we've seen historically. This just happens to be the Fiberite material here. (overhead) U.S. Poly is pretty much the same way. You can see that there is no general trends to the numbers, they are somewhat random. Part

of this I believe is differences in the size of the tag-end, permeability of the tag-end, and I understand that the original data was four hours then we moved it to 24 hours and sometimes in here (overhead) we moved back to 4 hours. I do not know the exact location where we changed our data base so there are different data bases within those numbers.

Group

Discussion

Don Beckley

It's a long time back in the 79 or 80's that it went to 24 hours and I think it was up there for quite a spell. I think most of that data is probably 24

John Koenig

Most of the data bases are 24 hours.

Group

Discussion

Don Beckley

This is part tag-end data?

Ben Neighbors

Yes, this is part tag-end data from actual hardware. I brought this (data) originally just to show that there is a reasonable amount scattered. Each point is an average of 7 data points.

Don Beckley

The one eight number was a summary that was taken shortly after STS8A that said this is what the total history was, kind of a thing like that so I assume this is that same data set.

John Koenig

Would that be 24 or 4?

Don Beckley

Most of that was 24 hour data by that time.

Pat Pinoli

Nothing that I have seen suggest that the data represents 24 hours. Twenty-four hours should get you up to the 2% and 3% range. This graph shows the effects of 4, 24 and 48 hours on residual volatile values.

Pat McGill

Keep in mind that this is tag-end data and the tag-end shift in 4 hour and 24 hour doesn't necessarily coincide with the

prepreg spec where we shifted from 4 to 24. I don't know how those things overlap.

Ben Neighbors I didn't even realize you had done a 24 hour test on your acceptance testing until about 10 minutes ago so I was not familiar when that shift in the data was made.

Don Beckley You want to run that down Ben just for everybodys edification.

John Koenig Can you find out which data that is and when the shifts took place? Can you determine the size of the residual volatile specimens?

Ben Neighbors Yes, I sure can.

John Koenig Is there a capability on the two dimensions on the specimens?

Ben Neighbors Probably not...

John Koenig There is just a weight number ?

Ben Neighbors Just a weight number. The other thing I was going to talk about was TGA and Karl Fisher. I can discuss some. I didn't know that you were going to be here Pat to collaborate on that. Kind of a blind presentation, I knew I was coming and I knew Bill was coming and that was it. The point that I was going to make is that 325°F you are roughly in this range (overhead-weight loss vs. temperature) and you are going to 500°F, you are just moving further down the curve. Whether this number in here (500°F) is more consistent than that number (325°F) I couldn't say. It is just a matter of which point you choose to accumulate your data base.

Pat Pinoli You have to arbitrarily pick a point. I choose a temperature consistent with the analytical device and prior to the resin pyrolysis temperature.

Ben Neighbors	Well I guess this is a Southern Research term right here. We've got the on-set of pyrolysis I guess at the time that it is 468°C. So that is kind of a moving target. (See TGA data in presentation)
Group	Discussion
Ben Neighbors	The next test that we ran was specific gravity. Our specification limits are 1.40 to 1.55 for the carbon and 1.7 to 2.15 glass phenolic and in all the phenolic data that I've looked at generally runned about 1.47 for the carbon. I believe that when I first saw this I believed we could tighten that band somewhat. Then when we ran our statistical analysis and looked at the data, we were all over the map. We found that hard to believe.
Group	Discussion
Ben Neighbors	The three sigma limits were wider than our current acceptance criteria. So we went and looked at some and decide why, what could be the cause. One of the things that we found was that we had, should be multiple test methods and not limits. We measured specific gravity by immersing in kerosene and water. We went ahead and decided to delete the water test methods, because generally they did not run the kerosene most of the time so there was some possibility for some shift in the data that way. Another thing they were using just a generic number for kerosene.
Ed Hemmelman	Why did we go from water to kerosene?
Don Beckley	We never did, we are still troubled by it.
John Koenig	Who's brilliant idea was that?
Ben Neighbors	There were some round robins, but I don't remember.

Pat Pinoli	<p>I ran a series of experiments at Palo Alto which addressed the issues. Specimens were machined square to provide a measured volume value. Previous studies have shown significant errors can be introduced into the measured volume; surface roughness tends to skew</p> <p>the values high and random error is introduced by non-parallel sides. The latter was found to be most pronounced problem in this method. Water has never been my choice as a displacement fluid due to the fiber characteristics and propensity of water to enter CCP voids.</p> <p>Water intrusion into voids has given us very erroneous data on the Trident II heatshield program. Parts rejected due to high density were found to actually be low on density, due to internal porosity. Our test results showed kerosene exhibiting the best specimen wetting, test accuracy and precision. Of course we measured the kerosene density prior to test, by Westphal technique.</p>
Ben Neighbors	That did match most of the data Sometimes they might use water
Don Beckley	Actually there is very little difference in the answer you get if you run both tests correctly.
Ed Hemmelman	Water is water except in different parts of the country use purification methods like chlorination. Kerosene is a generic term. I can't put a molecular formula on it.
Ben Neighbors	It comes off of the distillation column at a certain temperature ... specific gravity can vary quite widely.
Gary Hall	And for that we were directed by NASA.
Don Beckley	We never participated or heard about it, the globe was too late.

? Which is frequently the case, Don?

Don Beckley I guess so, unfortunately,

Ed Hemmelman Jon, were we given the opportunity to comment on the kerosene?

Jon Weispfenning Oh yea, we were given the opportunity to comment.

Bill Hall Wasn't Ben Goldberg the pusher all the way?

Group Discussion

Ben Neighbors So this type of variability when you look at it you say historically does not really mean anything. I think on U.S. Poly may show slightly more variation than Fiberite, but I don't understand why. Every number I have ever seen has probably been with the 1.46 to 1.49 range.

Don Beckley Are you talking about your scale or what: 45 1/2 to 47 plus is pretty tight numbering. If you specimens in and make measurements you don't repeat much better than that, in other words the test has enough error, Ben I was just commenting, I think that is good reproducibility. Okay, so it's even more averaged now.

John Koenig I think what you are more concerned about is limits on the glass phenolic.

Group Discussion

Ben Neighbors There is just a lot of variability. The next test that we run was a compressive test. Kind of a squash test. The specification limits for carbon are 18,000 and 55,000 psi and 15,000 and 65,000 for the glass phenolic. It is kind of hard to miss that with this material. The historical data for the carbon is running approximately 31,000 and the x-bar chart shows somewhat of a

variation but no explanation on this one. I think we've got some ideas.

Don Beckley

You probably had some specimens oriented edge wise.

Ben Neighbors

Test samples are .3 by .3 by .6 inches long and have no gauge section. Specimen orientation is defined as circumferential. Most of the time that will put you in a 45 degree warp filled orientation in a plane. On some parts it's the warp fiber direction, on other parts, it's the 45 DEG warp-fill orientation. The other thing is there nothing within the specification to prevent introducing an across-ply bias into the specimen. So you are testing essentially ... orientation. I guess if you wanted to continue with the test you need to better define what direction you are testing. The replacement tests we are looking into at Marshall to replace the present test includes a cross ply tensile and double notch shear. We are having problems with the specimen size on the across-ply tensile specimen and that is one reason for looking at the double notch shear specimen. Right now we really don't know which one we will use. When I say under development at Marshall on the double notch shear, that is Southern Research has been using the specimen for quite some time and MSFC working toward developing a data base for carbon phenolic.

Ed Hemmelman

What type of specimen is that double notch shear? Is it the same as this one or smaller?

Ben Neighbors

It is 1.25 inches long.

John Koenig

It is probably compression loaded isn't it?

Ben Neighbors

On the 4th test that we currently run was the resin content. The specifications for the carbon is 30 to 40 percent and 24 to

38 percent for the glass phenolic. The historical data for carbon is 34.4. For the glass it is 28.9 percent. Recently we've had problems with falling below 24 percent resin content. We were almost explicitly using Fiberite material and components and of course all the problems we have had we come to Fiberite material. Mainly because we haven't use any U.S. Poly. I'm going to elaborate on that more in a moment. As far as test methods, we used to have two test methods; one was the tube furnace method and there was a fisher burner method which was deleted in the summer of 1987. Most of the time MTI uses the tube furnace method. We deleted the Fisher method because we were just trying to reduce the number of variables within the testing program. One of the other things we are investigating is the ultrasonic resin content analyzer. According to the manufacturers it is 99 percent accurate and it takes about 5 seconds for the test. You can also perform, since it is a non-destructive test, on the actual hardware and not on the tag-end. Some of the problems that it is showing right now is apparently whenever you have a carbon and a glass substrate and you are trying to measure glass, with the carbon under the glass, it gives you funny numbers.

Jon Welspfenning

What are you using to investigate the ...?

Ben Neighbors

They are running a kind of round robin right now. They've sent some samples with a known-resin content. We ran those on the equipment. We are in a stage right now sending samples of unknown resin content to the vendor and having them tell MTI the resin values. The vendor will then send them back to MTI for testing.

John Koenig	What is the physics to what they are doing?
Ben Neighbors	I don't know.
Don Beckley	It would sound like it is the sonic modulus resin and the sonic modulus of a given fiber and then the velocity difference between the two is a ratio of the contents.
Group	Discussion
Don Beckley	You have got a third component and the distribution of it and...
?	It works good on more simple systems like an epoxy or graphite material, we are seeing it work quite well, but I don't know about the carbon...
Pat Pinoli	If you look at a typical microstructure for CCP, fiber, resin, and filler distribution, the prospect of obtaining meaningful data would appear low. In particular, the filler tends to segregate in random clumps within matrix regions.
Ben Neighbors	We are just looking at it to see what it is capable of, we don't have that defined yet.
Pat McGill	Who is the manufacturer?
Ben Neighbors	I don't know but I can find out. As I mentioned earlier, we are having problems with resin content in the glass phenolic. Originally we thought the problem was a combination of low resin content and then, within specification, and high flow within specification. We looked at our statistical data, the lot averages for the lots that had made bad components. The averages supported this theory. After that, I went back and looked at that specific role that went into a specific tag-end and it doesn't correlate anymore. That was kind of interesting. I am still a little bit puzzled about that. It really thought we understood it better.

Lou Ann Fikes Did you look at the certification data on that roll? You did not do test on that roll before they processed it.

Ben Neighbors All the rolls that made the bad parts were long gone.

Lou Ann Fikes And they don't normally do tests on rolls

Ben Neighbors No, we accepted it on the delivery data. I don't know maybe you (MSFC) have received a specific roll out of the lot that measures what you expect. What we've found since the investigation is that the tag-end design is fairly critical, we've had problems with the fixed housing, nose cap, forward nose ring, and throat.(Overhead) This is the fixed housing and nose cap and forward nose ring versus throat.

Don Beckley In each case you are talking about the glass behind it?

Ben Neighbors Yes, this is the glass over wrap. On these two components (fixed housing and nose cap) the glass goes on the mantle first, and then machined. And on these two you run the carbon first, machine the carbon, and over wrap the glass. What was unusual about those four components is that they all have free standing tag-ends and we've got some drawings that show you what I mean by a free standing tag-end. This is a throat ring. You can see the carbon wrap and then the glass over wrap and after they wrap the part they come down to the bottom (of the manual) and wrap a separate tag-end. This is a 1-1/2 inch bias glass cloth. We had up until last week, problems with the fixed housing, nose cap and throat, and then last week we lost our first forward nose ring. Most of the parts that have been scrapped have been associated with one lot of material.

Ed Hemmelman On the resin content test methods for carbon phenolic and glass phenolic the most producible one is the glass phenolic.

Ben Neighbors That's right it is a very clean test.

Ed Hemmelman You generally don't create resin in your parts. I guess my question is in a low resin content is there evidence of excessive resin bleed during the fabrication of parts?

Ben Neighbors That's essentially correct. It has to go somewhere. So, one of the things we looked at was tag-end sizes. Fixed housing was 2 inch tape, nose cap was 1-1/2 inch, nose ring has been 1-1/2 but has been changed to 2-1/2 and the throat ring is 1-1/2 inch tape. My original understanding was we were using 2-1/2 inch tape on the forward nose ring all of the time. We have lost about five fixed housings, two nose caps and two or three throats and no forward nose rings. We have to make more 2-1/2 inch. It turns out that we switched to 2-1/2 inch tape and the first part we made is the one part we lost. That was a surprise to me. However, the part that we did make that was bad came out of lot 5080 which probably gave us 3/4 of the bad parts. So there is something very unusual about that lot. One of the indications is the molecular weight is lower. We also had tag-end testing going on at Marshall Space Flight Center and we looked at the variation in resin content across the width of the tag-end and the first test we ran was on fixed housing which showed 27.49 percent resin content on the side of the specimen and 29.46 percent resin content in the center of the specimen, which did indicate a 2% variation. This is fairly important particularly if you are down on the 24-25% range of the specification. We ran another test on a throat ring which had some bias to it like 20.06 and 20.39. The variation wasn't

quite as broad, of course you might bring it closer together with a wider tag-end.

Ed Hemmelman You showed the range of trying to get from 24 to 32.

Ben Neighbors Let me keep going.

Ed Hemmelman I wonder how that range was ever established?

Ben Neighbors Beats me, we've done some testing and looked at low resin content as low as 16 percent and testing as high as 33 percent. So far the strongest ... is 16 percent.

Ed Hemmelman Ben, there is a way to calculate the theoretical weight percent based on optimum final resin and fiber in the composite and in glass phenolic, correct me if I am mistaken, but that number is closer to 20 percent than it is to 29 percent.

Ben Neighbors I guess you can become use to a number if everything is going fine, and you don't really look at it and all of a sudden you have a problem with low resin content. I made the mistake of assuming that 24 was a good number. Now I'm getting test data back and it isn't necessarily a good number. Shear properties, on a sample with 16% resin content, shear values went up by about 20 percent, tensile properties increased 100%. So I am afraid that we may have scrapped some of the best parts we ever made.

Don Beckley The one driving factor that enters it from the other side is that the tape wrapper wants a lot of glue to make his wrapping easier, so I think the tendency to have the number higher than the structural adequacy or even desirability comes from that.

Ben Neighbors I wasn't sure ten years ago what drove us in that direction and that may have been the key.

Don Beckley We share a mutual problem and it's more resin than the fabric wants to have and so we fight continually trying to distribute more resin than it wants.

Pat Pinoli What is the purpose of the glass over wrap? I have been trying to get a clear rationale for some time.

Ben Neighbors I've heard several theories. One, it is a better insulator, thermal conductivity in terms of heat You get twice the thermal capability as you do in carbon. And the other thing is you can prevent the carbon from leaching into the metal housing.

Pat Pinoli I thought the purpose initially was for structural support. I used that a couple times once was criticized that its intended useage was for thermal insulation.

Ben Neighbors It is starting to show up on the ASRM. It sure is expensive to put the glass on. It really is and you think carbon is \$50 a pound and glass is \$5 a pound so that is a good cost saving. But its not, because you have to go to another machine operation, another tape wrap and it is starting to show up.

Pat Pinoli Maybe Morton Thiokol could tell us what its intended use is?

Pat Doan The name on the print is insulator.

Ben Neighbors One thing we looked at is what kind of variables do we have in the manufacturing process. This turned out to be one of the startling things. We had been using three different separator film. We had a quarter inch on-center film, which is now deleted from the program, and we had a half inch on the center film is supplied by two vendors, and the whole size is 0.050/1000 and on half inch on the center for air tech material and 0.045 for the whole size of Richmond material and while they are both half inch on center, it turns out that the air tech has twice as many

holes as the Richman. And, in terms of bleader materials, we allowed mop tubing which I think has been shown to closes up when you apply pressure and you can't really breath through it. We have eighteen ounce polyester....

Pat Pinoli

Are they both qualified for all RSRM components?

Ben Neighbors

Yes. Always have been. Well I take that back, we deleted polyester in our infinite wisdom after STS8A on some components. We believe that the polyester is better than mop tubing. Of course in the prepreg material we use U. S. Poly and Fiberite. We have testing currently under way to test all of the described variables. We also have our standard test under way. We test resin content, residual vol, gravity, and progressive strength. Visual observations are Fiberite bleeds more than U.S. Poly, part of that reason is possibly because Fiberite does not have filler in their resin system, Poly does I understand. Polyester material bleeds slightly more than a mop tubing, it leaks a whole lot more but there really wasn't a whole lot of difference. As I was mentioning earlier the two ...1/2 inch separator films are not equal. The air tech has more holes and bleeds more resin into the bleeder. In terms of how much difference there is between the U. S. Poly and Fiberite, the U.S. Poly could cover anywhere from 1 to 5 percent of visual area resin bleeding into the separator films. The Fiberite material covers probably 85 to 95 percent, that's a significant difference in the capability for resin content in the cloth. Fiberite material will bleed through 4 or 5 layers.. So in terms of manufacturing there is a lot of variables going in. That is something we will probably have to address. One of the things

that is pretty interesting is just the difference in the tag-ends. Is the free-standing tag-ends that I was showing earlier are not representative of the parts basically is the bottom line. In fact it can have a significant effect on testing in terms of what are we really testing? We made need to go back in and redesign tag-ends.

Pat Pinoli Ben, would you go back to your diagram and show us what you mean by free standing.

Ben Neighbors This is a fixed housing. (Overhead) This is a tape mandrel surface here and this is the component in terms of the glass over wrap, it is the first one that was wrapped. Up here they come in and wrap a separate tag-end.

Pat Pinoli Okay, so it is a separate part. I wasn't aware that this technique was being employed.

Group Discussion

Pat Pinoli We all know how sensitive residual volatile desorption is to part thickness.

Ben Neighbors It is also sensitive to how far the resin staged and if the resin has a filler in it. The current resin content test does not give a true indication of how much the resin will flow when the component is cured. The four inch flow test gives the same resin flow number for both filled and unfilled resin systems and the components flow differently with the two resin systems. Apparently the component is at a temperature where the resin will flow for a longer time and you can really squeeze the resin out of it. It may also be a function of where the tag-end is in location to the vacuum port.

Pat Pinoli I assume that you are looking at a method whereby you can introduce the tag end into the component somewhere.

Ben Neighbors We are trying to figure out what the best way to come up with a more representative tag-end that we've got. We've got so many variable going into it. We need to come up with a different tag-end for each part. Then we get back to our original question. Low resin content. So far that is good up to a certain point. Eventually you will go over a cliff. Essentially that is everything I have. Are there any questions.

Ronald Higgins On the bleeder, was that data regenerated along that...

Ben Neighbors Yes that was Morton Thiokol. Tom Odell did that.

Don Beckley Should we machine off the glass to start again.

Ben Neighbors You have to start over each time.

Don Beckley Yes, that's too bad.

Bill Hall Before we start to wrap up does anybody have any general comments they would like to make about the meeting as a whole. If any of you want anything. On your second sheet you have our name, address, and phone number and everything. We hope to get full participation out of you guys. This is your committee now, and it's serious. That is, NASA is going to refer to this committee as something that guided them in what they finally wind up with. So, we need your participation. One of the things that I will do also unless I hear no's is, I will send to each one of you in your expertise area, the general test we went over today that are run and ask you for general comments on them. Whether they are applicable, not applicable, whether it is a good test, bad test, or this sort of thing. Like, Polycarbon will get the one on carbon fabric, Fiberite, U.S. Polymeric will get the one

on prepreg, this sort of thing. If you want to pass it on to someone else, fine. We do need feedback on that to get a sense of direction. This, as most of you know, is a five year program. We are scheduled to meet twice a year. Funding upfront was for the first two years. We have money in the bank for the first two year on this. The contracts, the second round of contracts, should be let this year for those of you that are participating in the structural integrity program. If there is any questions any of you have please call me or Linda and we will take care of it.

Don Beckley

Did I understand this effort is funded out of the second Integrity program?

Bill Hall

Yes.

Don Beckley

So this was pre or was never was actually bid?

Bill Hall

Right, this is part of the reserved money they have. On that second round, if you went to the pre-proposal meeting, they had two sets of money, one was a set that was going to be sent out RFPs and everybody bid on it, and the second set of money was for continuation of the existing programs. My funding came out of the existing funds. Okay, the next question is when and where do we meet again. Does November suit everybody? How about the week of November 14th. Does anybody have any problems with that. Tuesday, November the 15th? I assume everybody is in agreement to coming back out here. Is that not true? Both Pat and Don have offered to host us one time so you have your choice of San Francisco or back here. Then the next spring we will use the other if it is all right with everyone.

Any problem. How about San Francisco then in November and back here in the spring.

Pat McGill I would like to get maybe a little mileage out of one of these meetings, that I think would be beneficial to everyone. The fact that I see our ... has set up their research facilities in Tempe, Arizona now and it is quite a multi-million dollar undertaking and if we can schedule one for our facility in the Tempe area, I think everyone would enjoy seeing it and seeing what they have.

Bill Hall Would spring or fall be better?

Pat McGill It doesn't make any difference, just as long as we have the notice. Don't do it in the summer! It does get a little warm there.

Don Beckley You are almost on November a year from now.

Pat McGill That's fine. I would like to point out that we certainly have much finer accommodations there than we do in this office and it will really be a first class show place for you to see. I'm not trying to upstage or do anything, I think our industry is something that we should be proud of and I like to show it off.

Pat Pinoli I would make a comment in regard to Bill's desire to have some, individual feedback. I do think that it is appropriate that those people that are identified as representatives from Fiberite and U.S. Polymeric make a response in writing as to what their concerns are on existing specifications. In such a way that Bill then has something in hand that he can start to compare input and somehow put these comments into a package that will give feedback back to Marshall. Hopefully, there is a consensus in some area and general agreement in others. Supporting documentation, as long as it is brief, should be submitted.

Pat McGill Should we just arbitrarily select the current SRM carbon phenolic specification and assign that as a action item, that two respective companies would ...

Pat Pinoli Bill, will identify specific test procedures for study and submit them to individuals for follow-up.

Pat McGill This was all said this morning when I wasn't here.

Pat Pinoli Yes, that is right.

Ed Hemmelman I have got some definite opinions about the current specifications, I won't go into detail. But I think this committee could do a great service to the industry by taking a very close look at the carbon phenolic spec and making it user friendly. I won't go into any comments right now, because I think it would be rather unfair.

Pat McGill I think the term user friendly means, the fabricator as well as the prepregger.

Ed Hemmelman I think there are a lot of areas that are of concern that are not necessarily addressed, places that need clarification. Believe it or not and I believe Don will attest to this, that the prepreggers pay a lot of attention to this spec. That is how we make our living, making materials to meet that spec. I think that we are both very proud of the fact that we think we do a pretty good job of doing that. I think if we could do a service to the industry by redoing the spec and making it much better than it is right now.

Bill Hall That is the purpose of the committee, I see no reason why we can't do that. We have NASA representatives and fabricators, we have everybody here and I am sure NASA would welcome this. That is why they established the committee. This is what they

want. We certainly appreciate Pat McGill hosting this and you are a gracious host.

Pat McGill

I am pleased to kick it off.

Bill Hall

Any more comments. I would like to express my gratitude for all of you agreeing to do this and I look forward to seeing you all and hearing from you all before November. Let's keep it active and keep it going. Thank you and we'll see you next time.

**Standardization of the Carbon-Phenolic and
Carbon-Carbon Materials and Processes**

NASA Grant No. NAG8-545

**National Aeronautics and Space Administration
George C. Marshall Space Flight Center, Alabama 35812**

Ronald L. Nichols, Technical Officer



OBJECTIVE

The objective of this project is to select a set of specifications for the materials and processing of carbon-phenolic and carbon-carbon composite materials which yields a predictable and reproducible product with the desired properties.

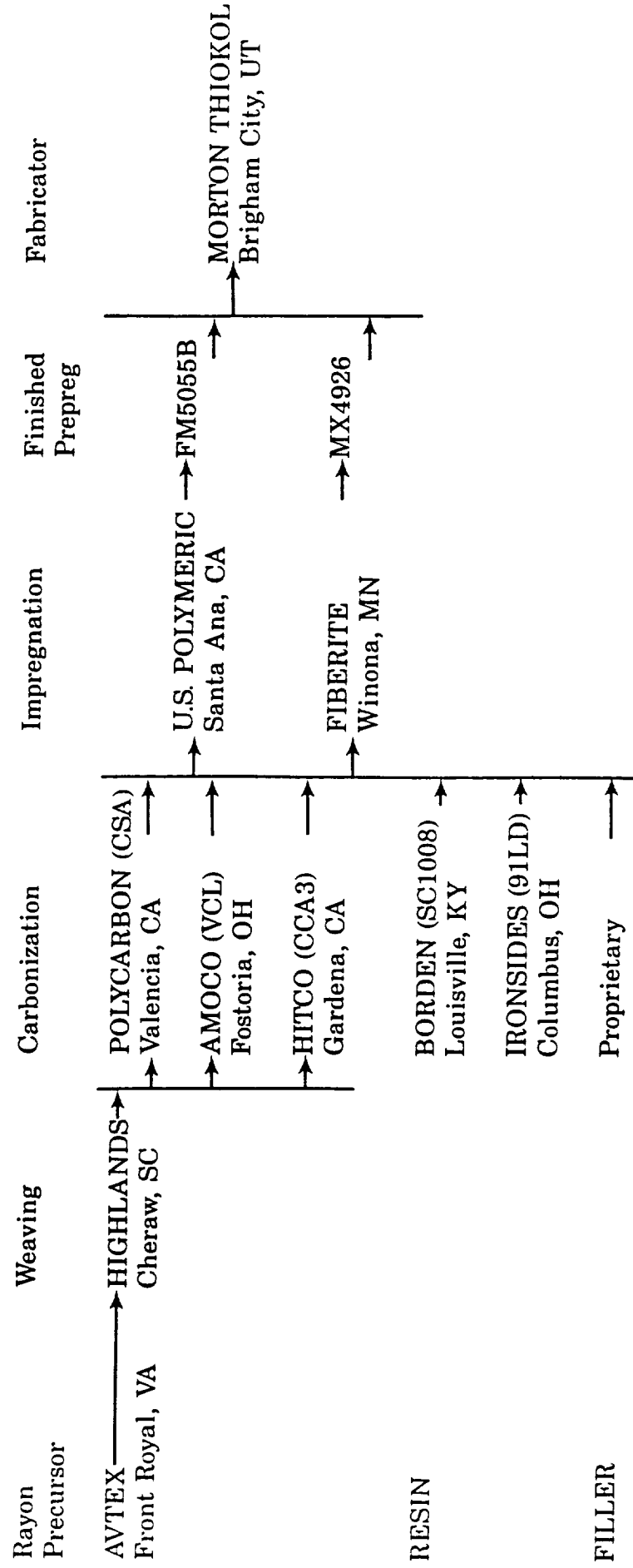
Objectives of Advisory Committee

Establishing and administering an industry-wide advisory committee that will meet twice a year to review, revise, add to/subtract from present specifications. The principal investigator will be responsible for setting up meetings, conducting meetings, and communications between committee members. He will also make up agendas and recommend priorities for the committee's consideration.

Criteria for Evaluating Goals of the Committee

1. Does the test method accurately measure the desired property?
2. Does this property yield useful information for the evaluation of the material to perform as required?

Materials Supplier Flow Sheet for Carbon Phenolic Nozzle Components



Properties Measured on Rayon Precursor

Rayon Fiber

Ash Content
Sulfur Content
Zinc Content
pH
Finish Extractables
Twist
Conditioned Strength
Conditioned Elongation
Moisture Content
Denier
Shrinkage

Rayon Cloth

Areal Weight
Thickness
Thread Count
 a. Warp
 b. Fill
Strength
 a. Warp
 b. Fill
Extractables
Denier
Moisture Content

Properties Measured on Carbon Cloth

Carbon Content

Ash Content

Moisture

Areal Weight

Breaking Strength

a. Warp

b. Fill

Specific Gravity

Thread Count

a. Warp

b. Fill

Thickness

Sodium Content

Properties Measured on Filler

Ash Content

Particle Size and Distribution

Chemical Analysis
Na, K, Ca, Li, Mg

TGA

Properties Measured on Resin

Resin Solids
Viscosity
Specific Gravity
Gel Time
Chang's Index
Sodium Content
pH
IR*
GPC*

Properties Measured on Prepreg

Cured (Panels)	Uncured
Specific Gravity	Cloth Content
Residual Volatiles	Dry Resin Solids Content
Compressive Strength	Carbon Filler Content
a. warp	Resin Flow
b. fill	Tack
Interlaminar Double	Sodium Content
Shear Strength	
Thermal Conductivity	
a. with ply	
b. across ply	
Coefficient of Thermal	
Expansion	
a. with ply	
b. across ply	
Flexural Strength	
a. warp	
b. fill	
Tensile Strength	
a. warp	
b. fill	

Properties Measured on Fabricator Cured Material

Specific Gravity

Ultimate Compressive Strength

Resin Content

Residual Volatiles

ASTM D1682 Breaking Load and Elongation of Textile Fabrics

All the possible combinations can be identified as follows:

Test Specimen	Type of Tester		
	Constant- Rate-of- Traverse G-T	Constant- Rate-of- Load G-L	Constant- Rate-of Extension G-E
Grab			
1-in. (25.4-mm) ravelled strip	1 R-T	1 R-L	1 R-E
2-in. (50.8-mm) ravelled strip	2 R-T	2 R-L	2 R-E
1-in. (25.4-mm) cut strip	1 C-T	1 C-L	1 C-E
2-in. (50.8-mm) cut strip	2 C-T	2 C-L	2 C-E
Modified Grab	MG-T	MG-L	MG-E

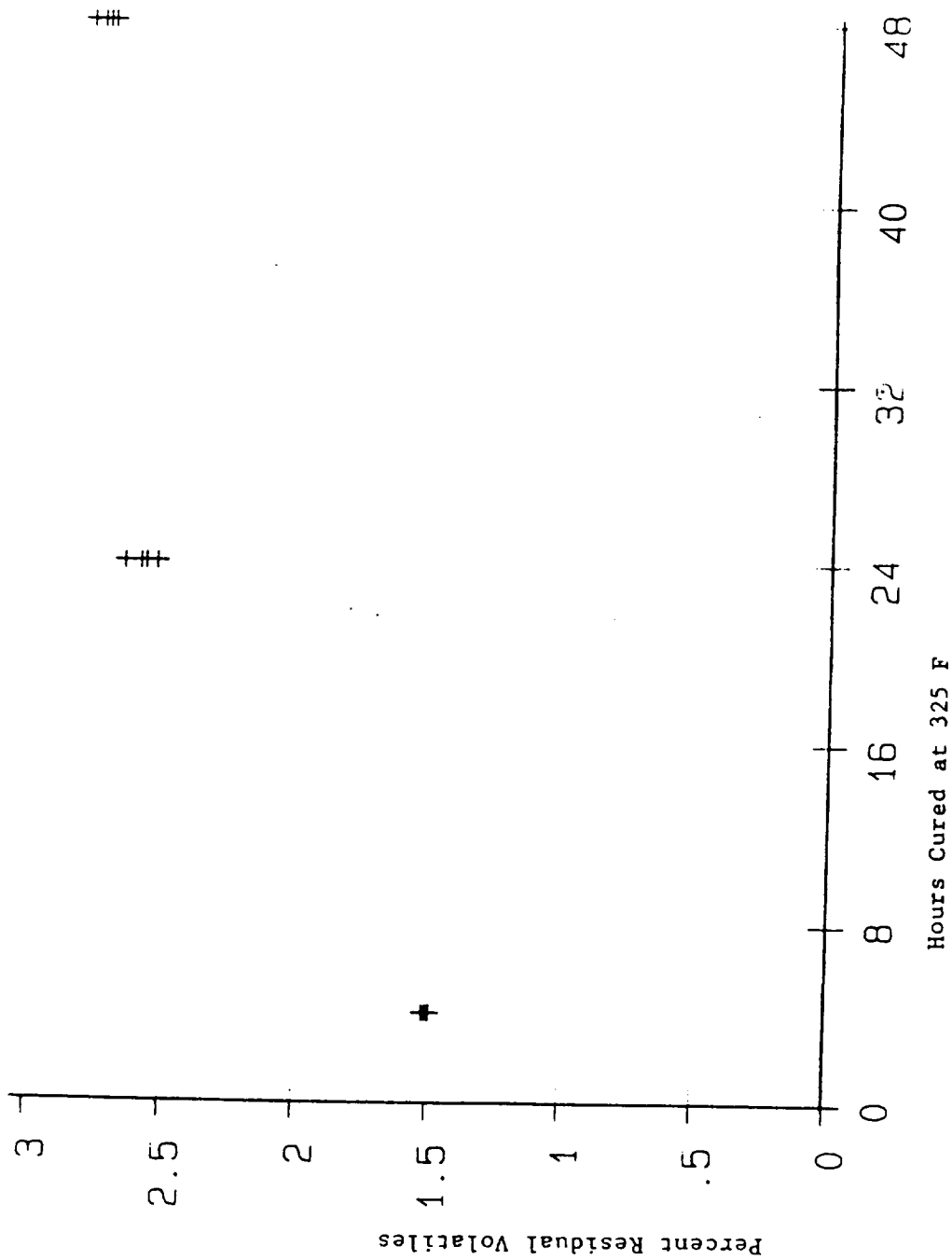
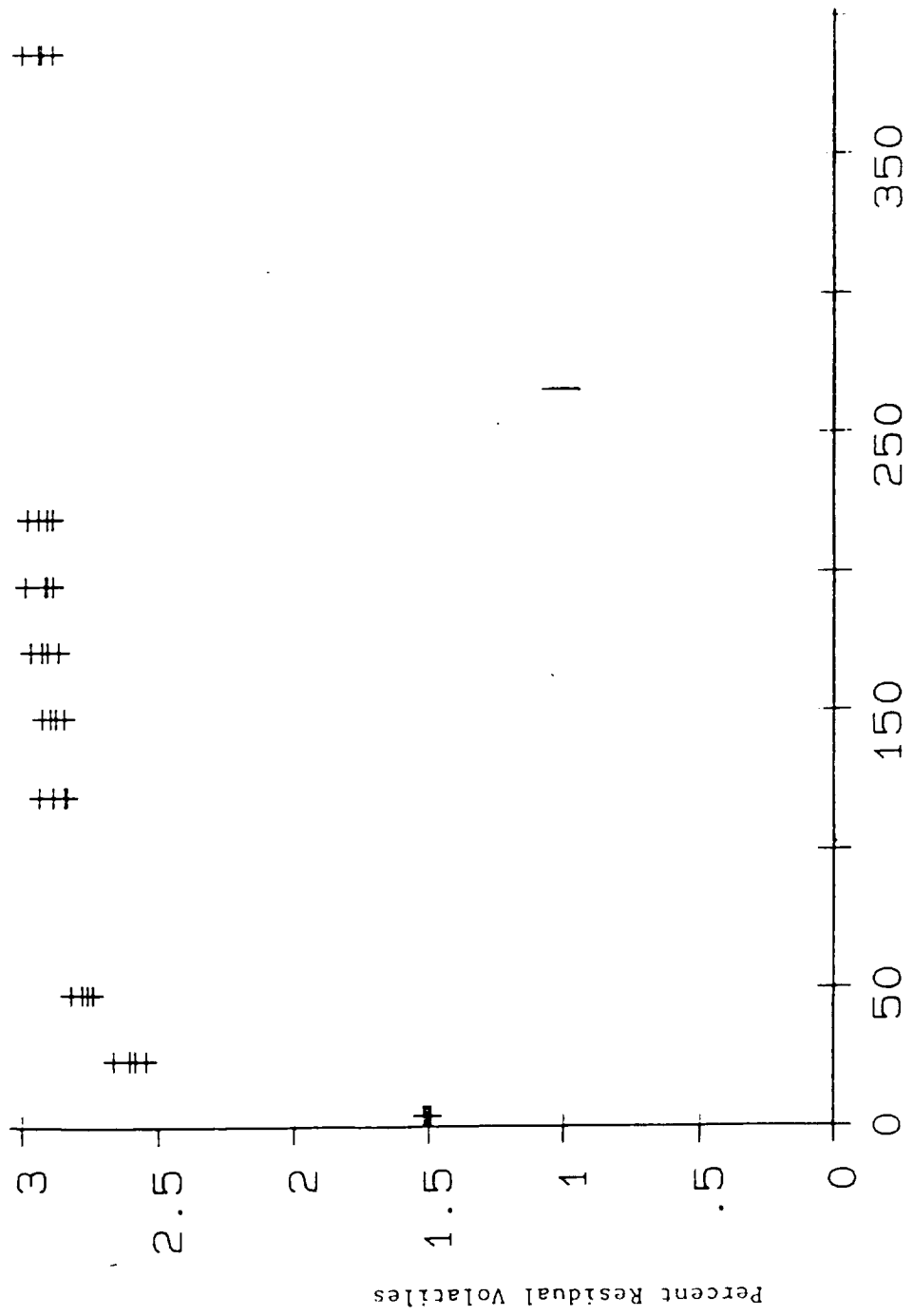
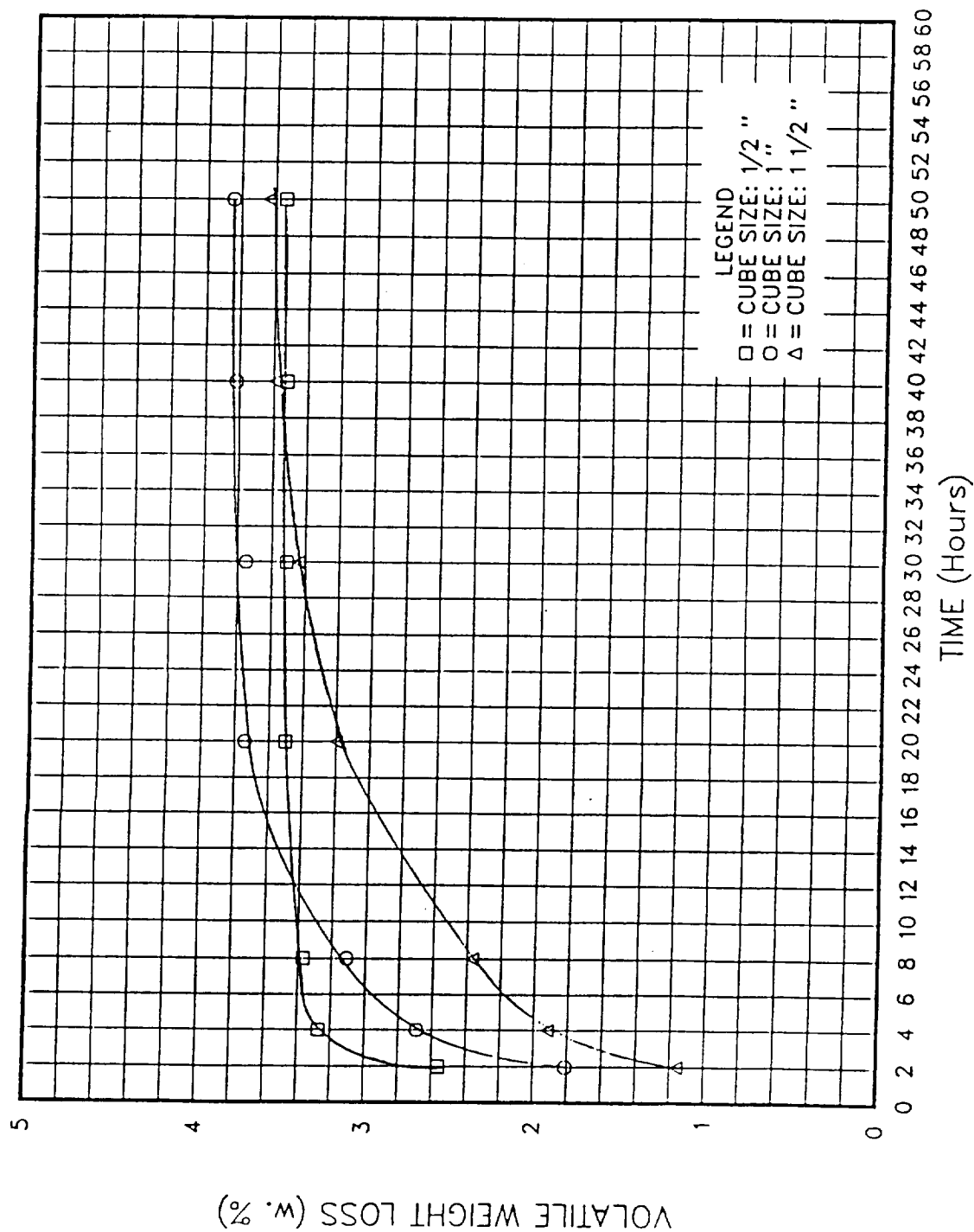


Figure 9. Percent Residual Volatiles versus Hours Cured at 325 F



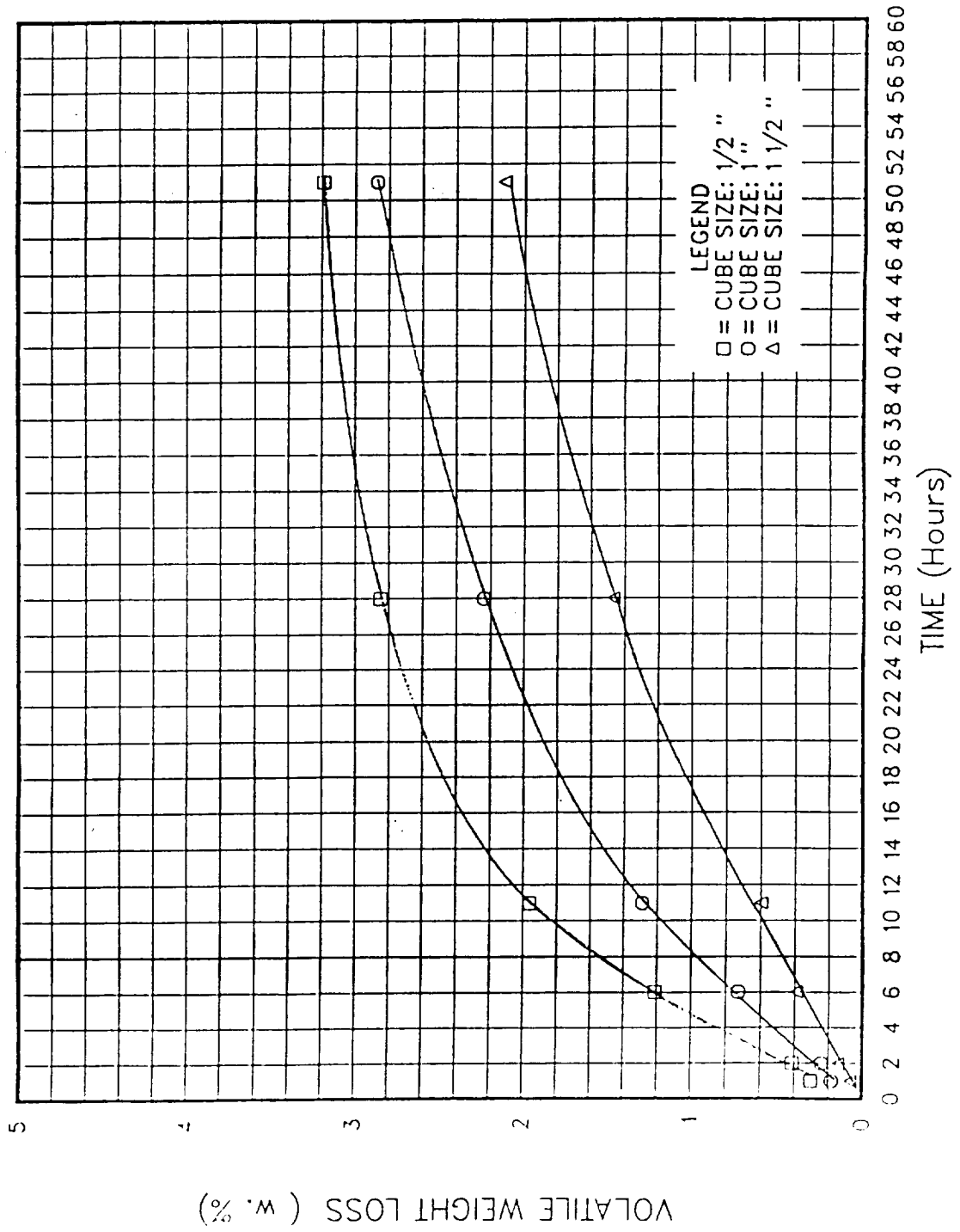
Hours Cured at 325 F
 Figure 10. Percent Residual Volatiles versus Hours Cured at 325 F

DESORPTION TIME DEPENDENCY STUDY
PANEL 8 CUBES DEVOL IN AIR CIRCULATING OVEN 325 F



DESORPTION TIME DEPENDENCY STUDY

PANEL 8 CUBES VACUUM DEVOL 220 F



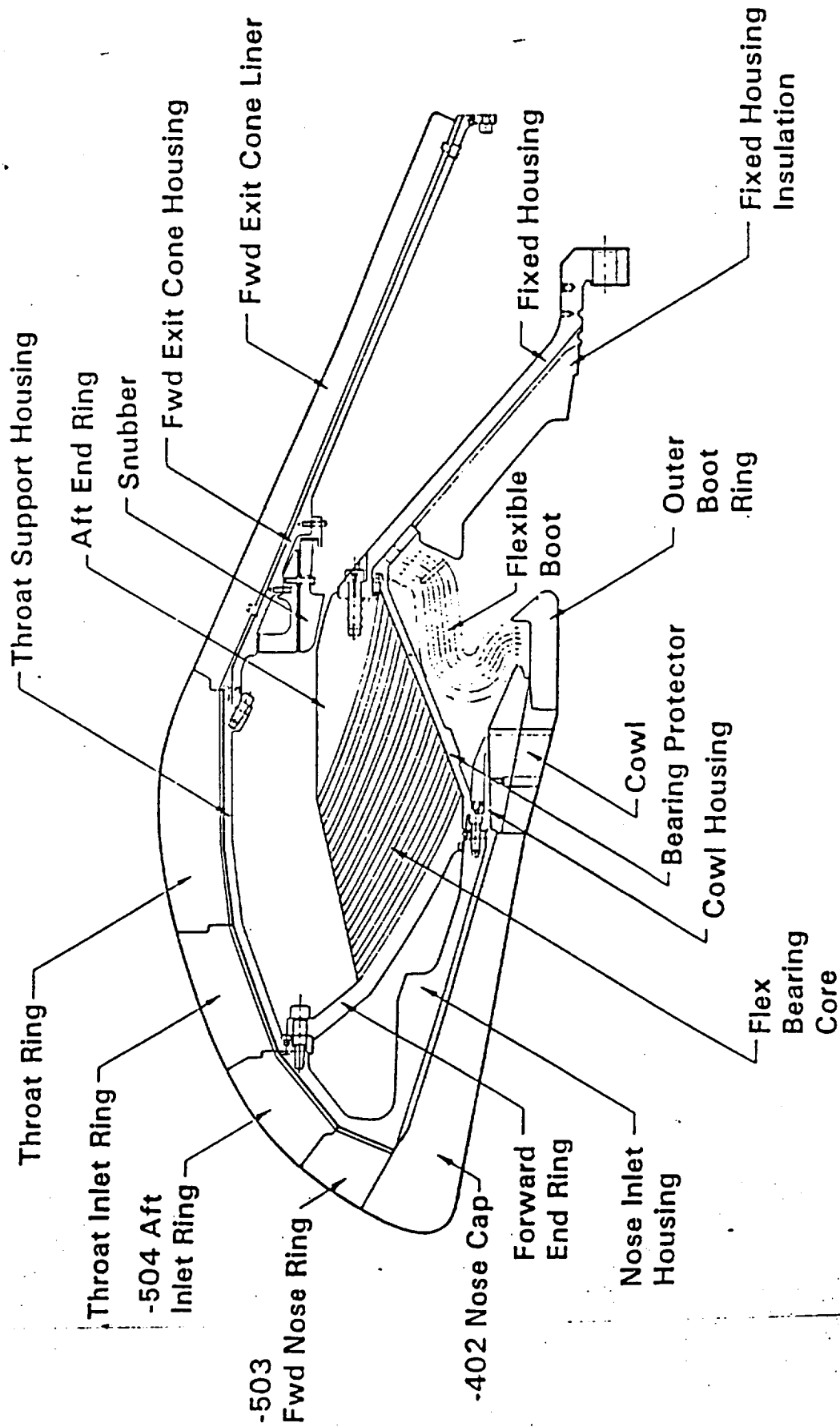
CARBON PHENOLIC TEST METHODS

LOW RESIN CONTENT IN GLASS PHENOLIC

BEN NEIGHBORS

MAY 3, 1988

RSRM Forward Nozzle Assembly



MORTON THIOKOL, INC.
Space Division

4006350 R1

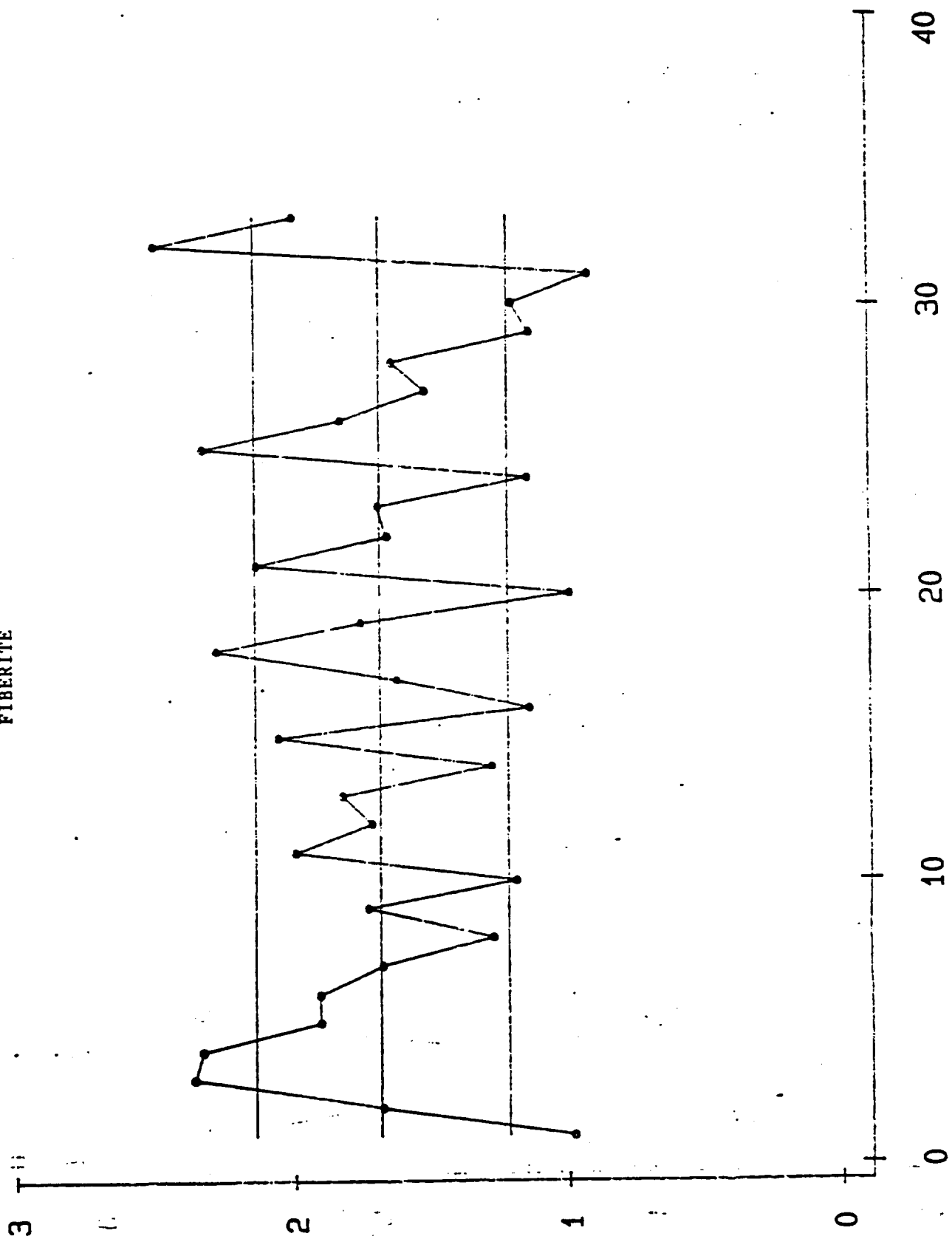
INFORMATION ON THIS PAGE WAS PREPARED TO SUPPORT AN ORAL PRESENTATION
AND CANNOT BE CONSIDERED COMPLETE WITHOUT THE ORAL DISCUSSION

CARBON PHENOLIC MATERIAL TESTING

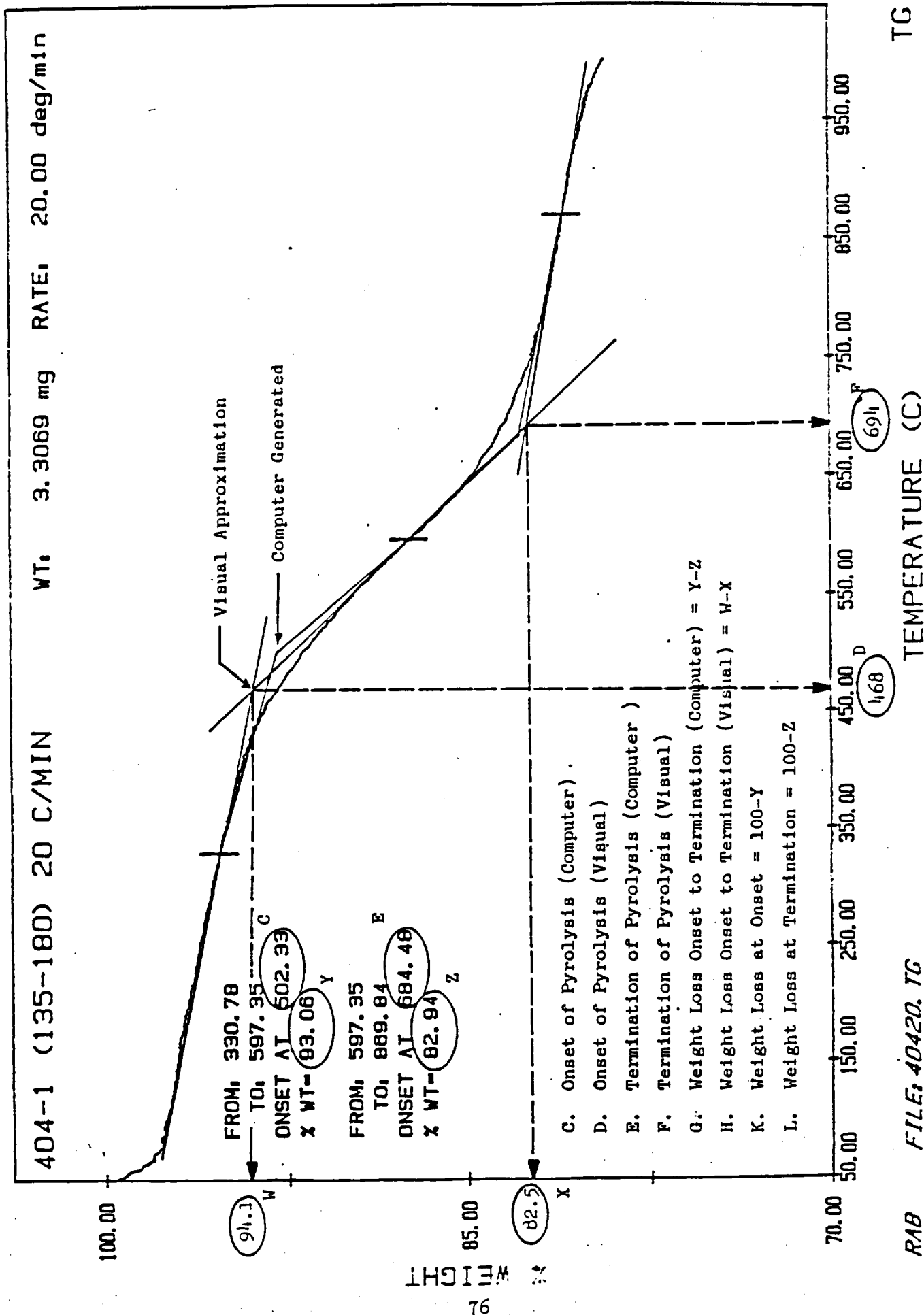
CURRENT RSRM TAG END TESTING

1. RESIDUAL VOLATILES
 - A. SPECIFICATION LIMITS
 - I. CARBON PHENOLIC 0 - 3%
 - II. GLASS PHENOLIC 0-3.25%
 - B. THE HISTORICAL DATA
 - I. CARBON 1.8%
 - II. X BAR CHART
 - C. TGA
 - D. KARL FISHER TEST
 - I. POSSIBLE REPLACEMENT TEST

FIBERITE



X-BAR Chart for Volatiles, Carbon



TG

RAB FILE: 40420. TG

DATE: 87/09/24 TIME: 12.42

Figure 3.6-2. TGA Demonstration Graph

CARBON PHENOLIC TEST METHODS

2. SPECIFIC GRAVITY

A. SPECIFICATION LIMITS

I. CARBON PHENOLIC 1.40-1.55

II. GLASS PHENOLIC 1.70-2.15

B. HISTORICAL DATA

I. CARBON 1.463

II. X BAR CHART

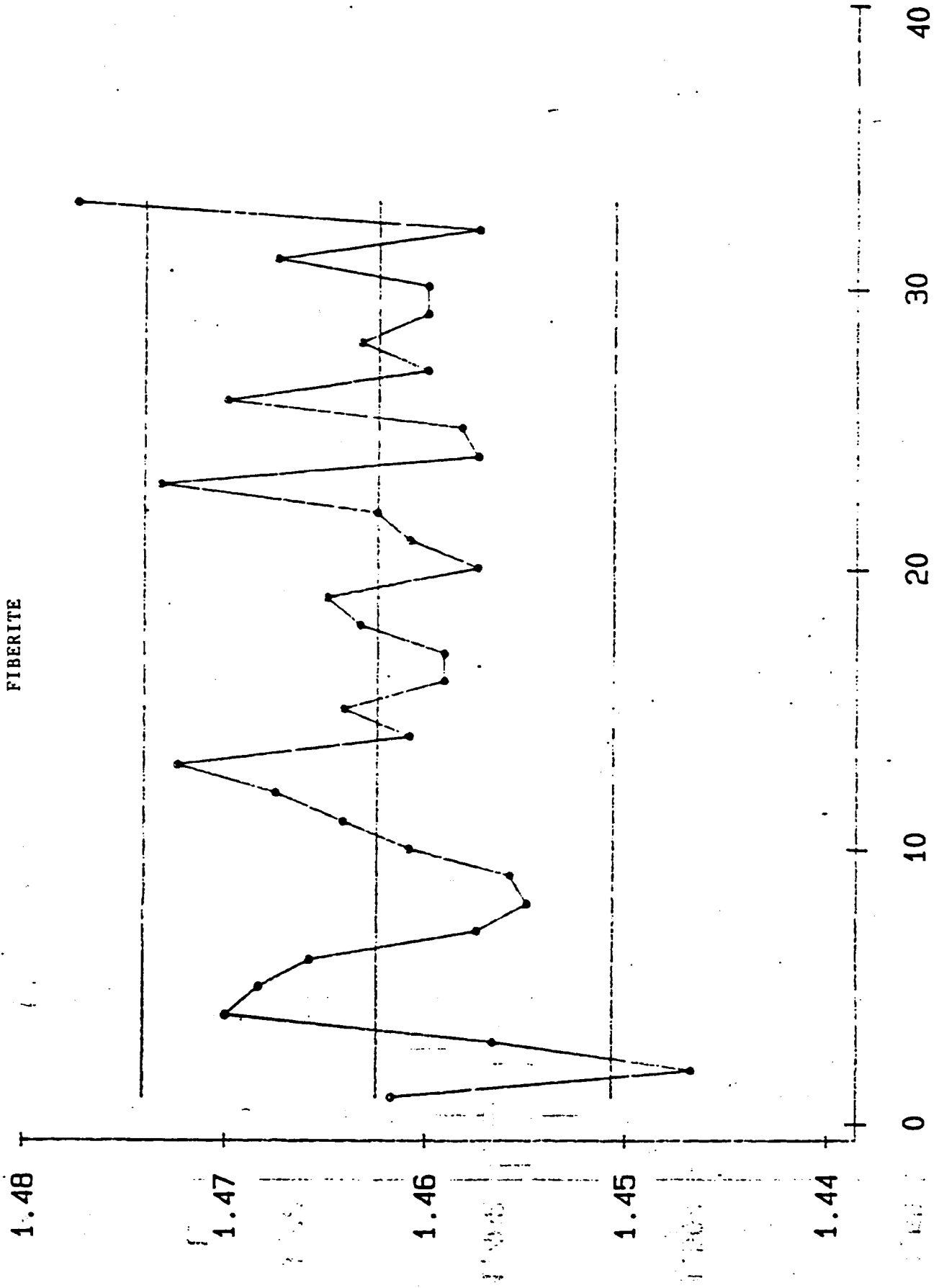
C. MULTIPLE TEST LIMITS

I. KEROSENE

II. WATER - DELETED SUMMER 1987

D. SPECIFIC GRAVITY OF EACH LOT OF KEROSENE NOW TESTED

FIBERITE



CARBON PHENOLIC TEST METHODS

3. COMPRESSIVE STRENGTH

A. SPECIFICATION LIMITS

I. CARBON PHENOLIC 18,000 PSI TO 55,000 PSI

II. GLASS PHENOLIC 15,000 PSI TO 65,000 PSI

B. HISTORICAL DATA

I. CARBON 31,000 PSI

II. X BAR CHART

C. TEST SAMPLES

I. SIZE 0.30 x 0.30 x 0.60 INCHES

II. NO GAGE SECTION

D. SPECIMEN ORIENTATION - CIRCUMFERENTIAL

I. 45 DEGREES WARP - FILL (IN-PLANE)

II. WARP FIBER DIRECTION

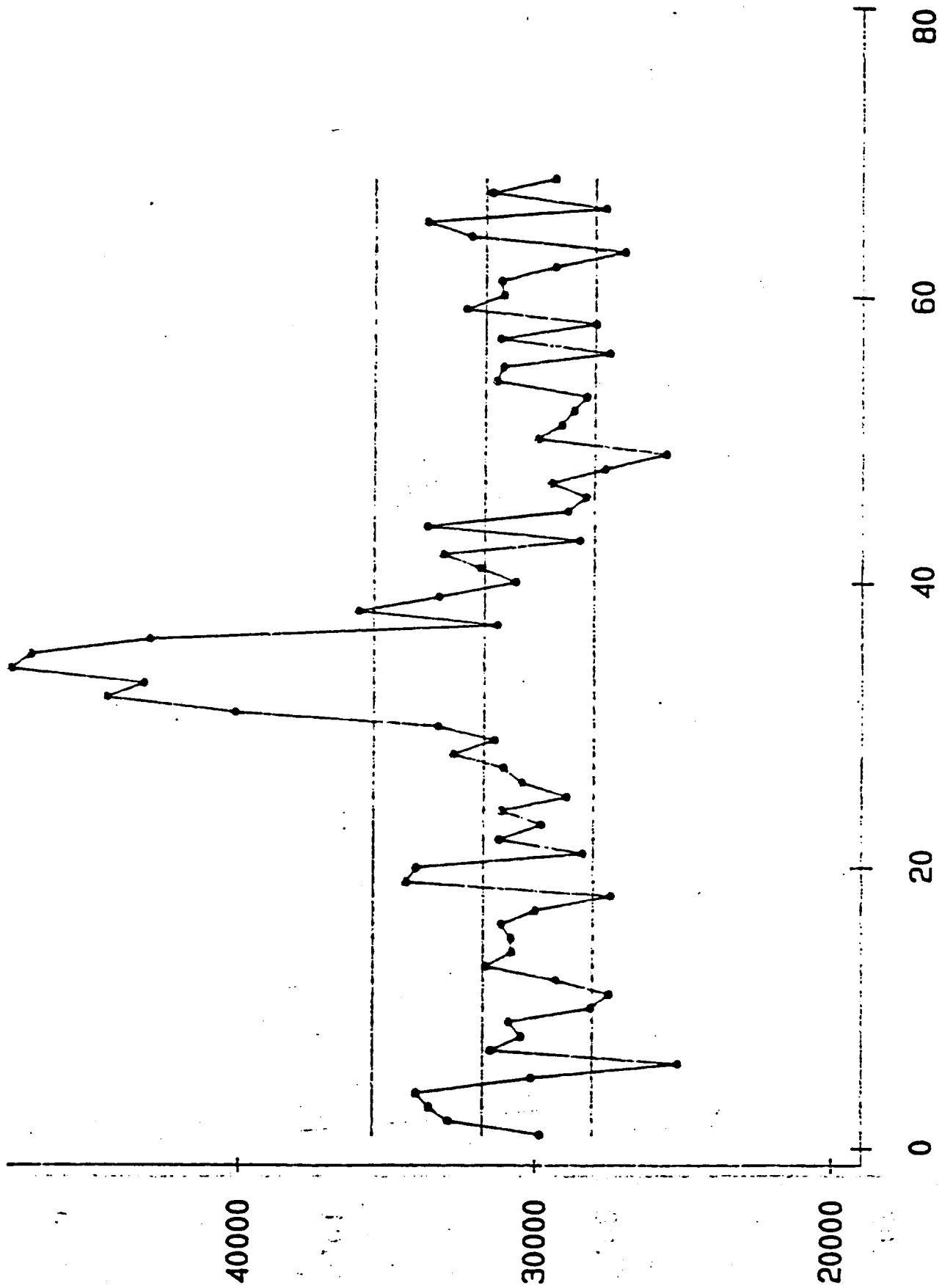
III. POSSIBLE CROSS PLY BIAS

E. REPLACEMENT TEST UNDER DEVELOPMENT

I. ACROSS PLY TENSILE

II. DOUBLE NOTCH SHEAR - DNS

III. CURRENTLY UNDER DEVELOPMENT AT MSFC



CARBON PHENOLIC TEST METHODS

4. RESIN CONTENT

A. SPECIFICATION LIMITS

I. CARBON PHENOLIC 30-40%

II. GLASS PHENOLIC 24-38%

B. HISTORICAL DATA

I. CARBON 34.4%

II. X BAR CHART

III. GLASS 28.98%

C. TEST METHODS

I. TUBE FURNACE METHOD

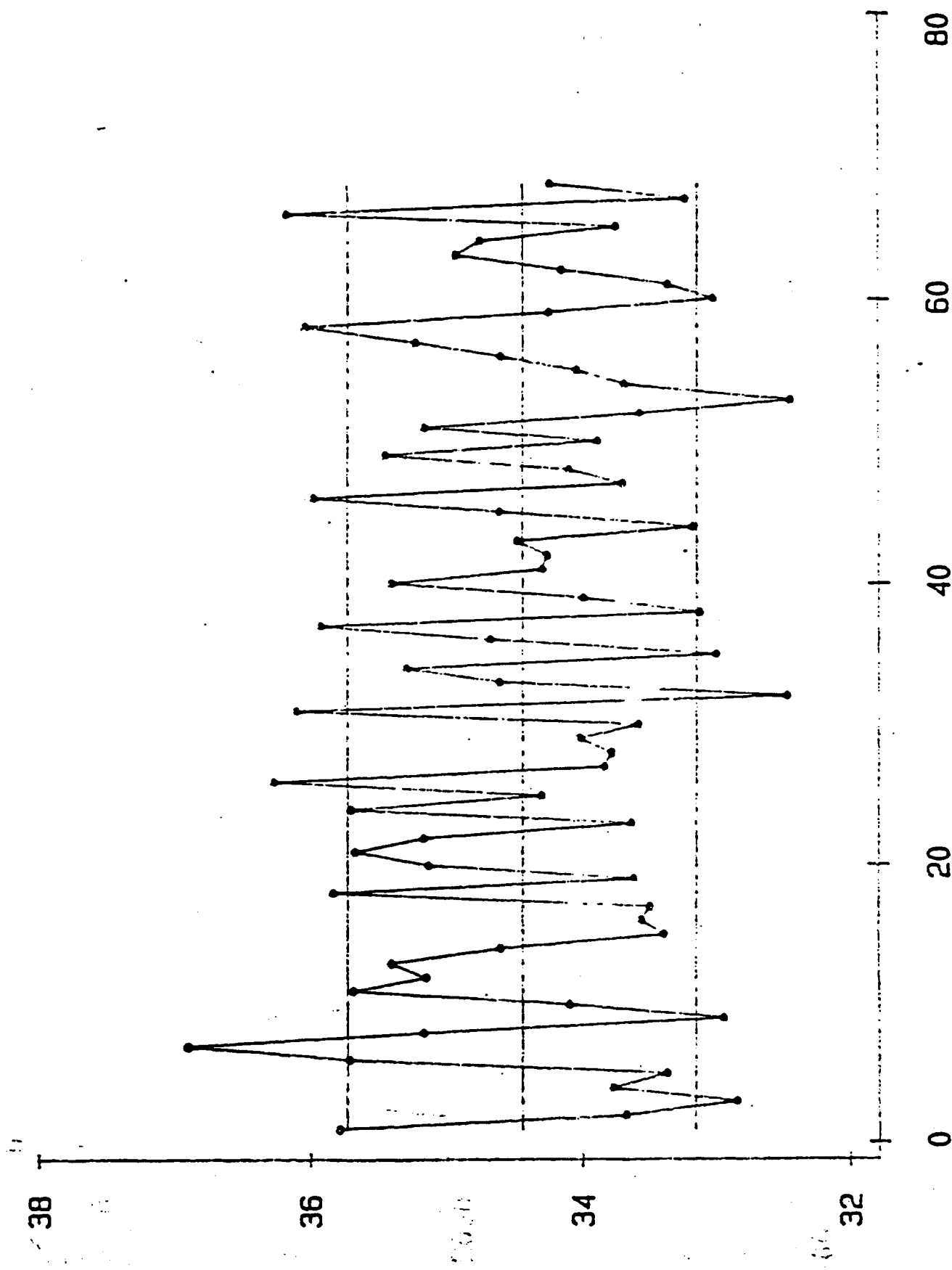
II. FISHER BURNER METHOD -
DELETED SUMMER 1987

D. INVESTIGATING ULTRASONIC RESIN CONTENT
ANALYZER

I. 99% ACCURATE

II. 5 SECOND TEST

III. NON DESTRUCTIVE - CAN TEST COMPONENT



X-BAR Chart for Resin Content, Carbon

LOW RESIN CONTENT IN GLASS PHENOLIC

1. RECENT PROBLEM WITH LOW RESIN CONTENT

A. PROBLEM WAS ORIGINALLY THOUGHT TO BE A COMBINATION OF LOW RESIN CONTENT (WITHIN SPECIFICATION) AND HIGH FLOW (WITHIN SPECIFICATION)

I. LOT AVERAGES FOR RESIN CONTENT AND RESIN FLOW SUPPORTED THIS

II. ROLL AVERAGES FOR RESIN CONTENT AND RESIN FLOW DO NOT SUPPORT THIS

B. TAG END DESIGN CRITICAL

I. ALL OF THE LOW RESIN CONTENT PROBLEMS HAVE BEEN ASSOCIATED WITH 4 COMPONENTS

FIXED HOUSING
NOSE CAP
FWD NOSE RING
THROAT

II. ALL 4 COMPONENTS HAVE FREE STANDING TAG ENDS

LOW RESIN CONTENT IN GLASS PHENOLIC

2. TAG END

- A. FIXED HOUSING 2.0 INCH TAPE
- B. NOSE CAP 1.5 INCH TAPE
- C. FWD NOSE RING 1.5 INCH TAPE
2.5 INCH TAPE
- D. THROAT RING 1.5 INCH TAPE

3. MSFC TAG END TEST RESULTS

- A. VARIATION IN RESIN CONTENT ACROSS THE WIDTH
OF TAG ENDS

	<u>SIDE</u>	<u>CENTER</u>
FIXED HOUSING	27.49%	29.46%
THROAT RING	20.06%	20.39%

LOW RESIN CONTENT IN GLASS PHENOLIC

5. TESTS- RESULTS OF RESIN CONTENT VARIABLES

A. RESIN CONTENT, RESIDUAL VOLATILES, SPECIFIC GRAVITY AND COMPRESSIVE STRENGTH TESTS ARE UNDER WAY

B. VISUAL OBSERVATION

I. FIBERITE BLEEDS MORE THAN US POLY

II. POLYESTER BLEEDER MATERIAL BLEEDS SLIGHTLY MORE THAN MOP TUBING

III. TWO VENDORS OF 1/2" PREPARATED FILMS ARE NOT EQUAL

AIRTECH WL-4500 HAS MORE HOLES AND BLEEDS MORE RESIN THAN RICHMOND E-3760